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INVESTIGATING THE USE OF DIFFERENT TYPES OF SEALANT MATERIALS  
FOR CEMENT REMEDIAL APPLICATIONS

by

MURAD MOHAMMEDAHMED ABDULFARRAJ

A DISSERTATION

Presented to the Faculty of the Graduate School of the  
MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

DOCTOR OF PHILOSOPHY

in

PETROLEUM ENGINEERING

2019

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## **PUBLICATION DISSERTATION OPTION**

This dissertation consists of the following four articles, formatted in the style utilized by Missouri University of Science and Technology:

Paper I: Pages 10 - 36, has been accepted as a journal paper by the Journal of Petroleum Exploration and Production Technology under the title “The Potential of Using Micro-Sized Crosslinked Polymer Gel to Remediate Water Leakage in Cement Sheaths.”

Paper II: Pages 37 - 63, will be submitted to the Journal of Petroleum Exploration and Production Technology “The Application of Micro-Sized Crosslinked Polymer Gel for Oil Control in Channeled Cement Cores.”

Paper III: Pages 64 - 85, will be submitted to the Journal of Petroleum Exploration and Production Technology “The Application of Epoxy Resin Sealant to Remediate Water and Oil Leakage in Cement Fractures and Channels.”

## ABSTRACT

The overall objective is to investigate the ability of several material to mitigate cement failures such as channels, fractures, and micro-annuli in oil and gas wells. The sealant material that can also be used in the wells that are used for geologic storage of carbon dioxide CO<sub>2</sub>. Evaluating the sealant material is critical to improve the zonal isolation of the wells, which would lead to safer operations. The approach of this dissertation is to utilize particles gel that has been used successfully in conformance control application, and epoxy resin systems, that have superior seal characteristics compared to conventional Portland cement. The specific goals are: first, to investigate the sealants that are suitable for the expected wellbore environments and have acceptable viscosity, high bond strength to casing, high injectivity, low permeability, high ductility and high compressive and tensile strength. Secondly, to evaluate the effectiveness of these materials to repair cement's flaws, in the lab, by using cement cores flooding experiments. Two approaches will enable these objectives: 1) performing intensive laboratory measurements to investigate the rheological behavior and mechanical properties of the particles gel and epoxy sealants 2) conducting intensive core flooding experiments to determine sealant material injectivity and determine the seal plugging efficiency to the water and oil leakage from cracked and fractured cements. The outcomes of this study show the epoxy resin sealant develops good bonding strength with the cement but the particles gel does not. The epoxy resin sealant was able to completely prevent oil and water leakages while the particles gel was not able to achieve that.

## ACKNOWLEDGEMENTS

In the name of Allah, the most Beneficent, the most Merciful.

I would like to express my sincere gratitude to King Abdulaziz University (KAU) and Saudi Arabian Cultural Mission (SACM) for rewarding me full funded scholarship and for their friendly assistance throughout the study.

I would like to gratefully thank my advisor, Dr. Abdulmohsin Imqam for approving me to join his research group, and for his encouragement, inspiration, and critical comments.

I would like to thank my PhD committee members, Dr. Shari Dunn Norman, Dr. Mingzhen Wei, Dr. Kelly Liu, and Dr. Ahmed Algarhy for their time and efforts in examining the dissertation and all the constructive feedback.

I would to acknowledge my research group members for their great help and valuable discussions especially Mohammed Alkhamis.

I want to express my sincere gratitude to the rock mechanics and explosives research center.

Last but not least, a great thanks to my family. Words cannot express how grateful I am to them for all the support and encouragement.

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## **1. INTRODUCTION**

### **1.1. STATEMENT AND SIGNIFICANCE OF THE PROBLEM**

Cementing is a major step in the construction and sealing of oil and gas wells. During the life of the well, cement is prone to cracking due to change in downhole conditions. The cement integrity must not be compromised at any time during drilling or production operations. If the cement is not properly completed and abandoned, it may develop leakages during any stage of the life of the well (Watson and Bachu, 2009). Fluids (water or hydrocarbon) can migrate through pathways within the cement itself or between the cement and its surroundings. When the wellbore integrity is compromised, these pathways occur and formation fluids are allowed to migrate between formations and/or from the formation to the surface. Leakage of water, gases and hydrocarbon fluids through cement pathways may occur during the drilling stage of the well, production stage, or after the abandonment of the well, which may endanger personnel and the environment (Davies et al., 2014; Alkhamis and Imqam 2018; Ahdaya and Imqam, 2019; Al-Hameedi et al., 2019). The leakage of fluids along the interface between the wellbore and formation is a primary concern in hydrocarbon recovery (Dusseault et al., 2000). Leakage pathways in the cement annulus can happen due to mechanical failures as a result of pressure and temperature cycles, chemical degradation due to the corrosive formation fluids (Zhang and Bachu, 2010), or due to improper slurry design. Improper slurry design includes the use of slurries with low densities, which may allow formation fluids to create channels within the cement sheath, the use of slurries with high fluid loss, which may affect the mechanical properties of the set cement, and the use of rigid cement, which can get fracked as a result

of casing expansions and contractions. In addition, failures can occur due to improper mud removal. The leftover of mud on the surface of the formation and casing can prevent the cement from bonding with its surroundings, creating micro-annuli. These failures and more can happen, creating pathways for formation fluids through the cement barrier.

Commonly, cement squeeze remedial operation is performed to seal cement leakages. However, cement squeeze in certain situations cannot be used effectively to mitigate and prevent the leakage because of low cement injectivity, pressure restriction, pin-hole leakage, micro-channels and fractures inside leaked formation, and micro-cracks within primary cement (Jones et al. 2014). Micro fine cement is intended to increase penetration, however, the fundamental problem of adhesion versus cohesion bond failure of cement and steel casing in the downhole environment is not addressed by most, if not all, current repair materials. Adhesion failure is characteristically brittle and unpredictable classical cement-steel failure is a typical adhesion failure. When special cements that form a strong bond to steel are used, partial cohesion failure may occur. The very low fracture toughness of cement plays a significant role in cohesion failure. To overcome these limitations, several types of sealants, such as polymer gel and epoxy resin, can be used to plug the pathways created in the cement.

This research investigates the use of micro-sized cross-linked polymer gel and epoxy resin as sealant materials to mitigate cracked cement sheaths and significantly achieve higher bond strength between the sealant and its surroundings.

## **1.2. EXPECTED IMPACTS AND CONTRIBUTIONS**

Results obtained from this research will determine the potential of using micro-sized cross-linked polymer gel as a sealant material to mitigate the cracked cement sheaths.



Therefore, an experimental setup was designed to investigate polymer gel capability to prevent fluids leakage through channels in the cement. To the authors' knowledge, very little experimental work has been conducted to investigate the use of crosslinked micro-gel in cement zonal isolation. In addition, this study will provide petroleum engineers with a fundamental understanding about using Novolac epoxy resin sealant for wellbore integrity applications. This work will study the impact of temperature on the curing time of the sealant, the ability of the sealant to plug the pathways, and the ability of the sealant to stop leakages. The lab experiments incorporate rheological examination, curing time estimations, blocking efficiency, and mechanical properties.

### **1.3. OBJECTIVES**

The primary objective of this research is to investigate the use of micro-sized cross-linked polymer gel and epoxy resin as sealant materials to mitigate cracked cement sheaths and significantly achieve higher bond strength between the sealant and its surroundings. To gain a better understanding of the mechanism, process, and performance of micro gel extrusion through cement fracture, extensive core flooding experiments will be performed. Through this research activity, a specific objective can be obtained:

- ❖ To investigate the rheological behavior and mechanical properties of the particles gel and epoxy sealants.
- ❖ To determine what factors impact polymer gel injectivity through the cracks and fracture cement and identify polymer gel injectivity mechanisms.
- ❖ To determine what factors impact the plugging efficiency to water and oil leakages from cement's fractures and channels using particles gel and epoxy sealant materials.
- ❖ To identify which sealant material is the most applicable for wellbore integrity.

Based on these objectives, this research will provide a comprehensive knowledge of the micro gel injectivity mechanisms, plugging performance, compared to epoxy resin materials.

#### 1.4. SCOPE OF THE WORK

This research is primarily a laboratory study to investigate factors affecting the micro gel propagation and plugging efficiency and compare the gel's performance to that of the epoxy resin intended for cement fractures and channels. Core flooding experiments will assist in understanding the prevailing mechanism and performance of micro gel propagation and epoxy resin through these voids. Figure 1.1 shows this research scope of work, which includes the main required experiments to accomplish the research objectives. Experiments required for understanding the extrusion and plugging efficiency of micro gel and epoxy resin are included in the scope of work.

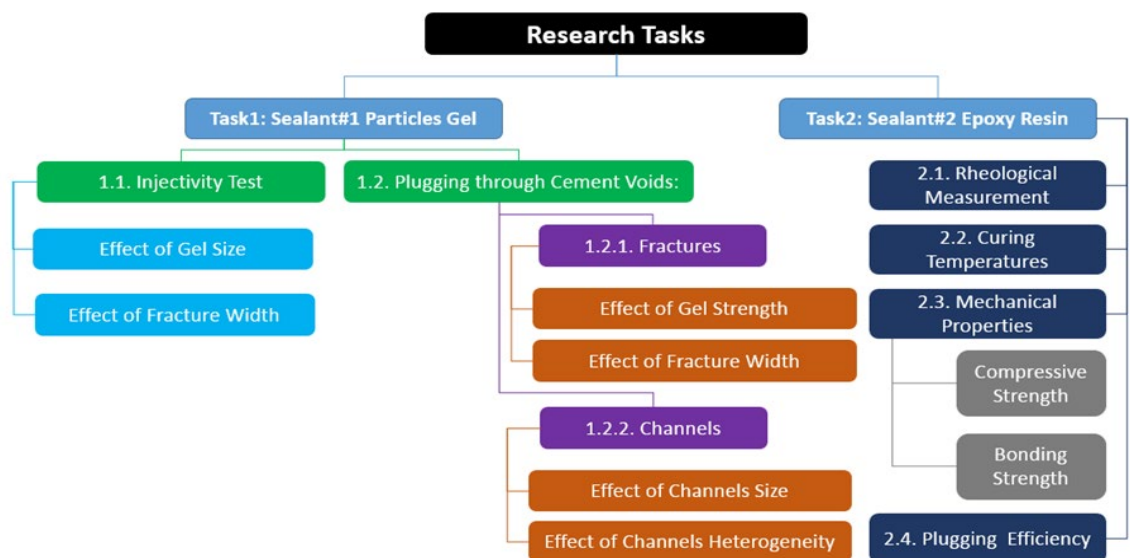


Figure 1.1 Research Scope of Work

## **2. BACKGROUND AND EXISTING TECHNOLOGIES**

### **2.1. WELLBORE INTEGRITY FAILURES**

Wellbore integrity can be defined as the ability of the wellbore cement to maintain isolation between permeable reservoirs and impermeable layers in geological formations. Wellbore cement often develops leaks during the life of the well if not properly completed and abandoned (Watson and Bachu, 2009). When wellbore integrity is compromised, leakage occurs and pressurized fluids are allowed to migrate vertically to other formations and/or to the surface. Leakage of gases and hydrocarbon fluids through wellbore cement can occur during drilling, hydraulic fracturing, production, or after abandonment of the well, which may endanger the health and the safety of the field workers and the environment (Davies et al., 2014). Specifically, the highest probabilities of leakage are associated with decommissioned wellbores in comparison to wells associated with producing or injecting.

The potential leakage of fluids along the interface between a wellbore and formations is a primary concern in hydrocarbon recovery (Dusseault et al., 2000) and carbon sequestration (van der Tuuk Opedal et al., 2013). Leakage pathways in the cement annulus can be generated due to either mechanical well failures from cyclic pressure and temperature changes as shows in Figure 2.1, or chemical degradation from corrosive formation fluids (Zhang and Bachu, 2010). In the case of carbon dioxide (CO<sub>2</sub>) storage, CO<sub>2</sub> can potentially leak through the fractures and channels developed in the cement or micro annuli formed between the cement and its surroundings (Watson et al., 2007).

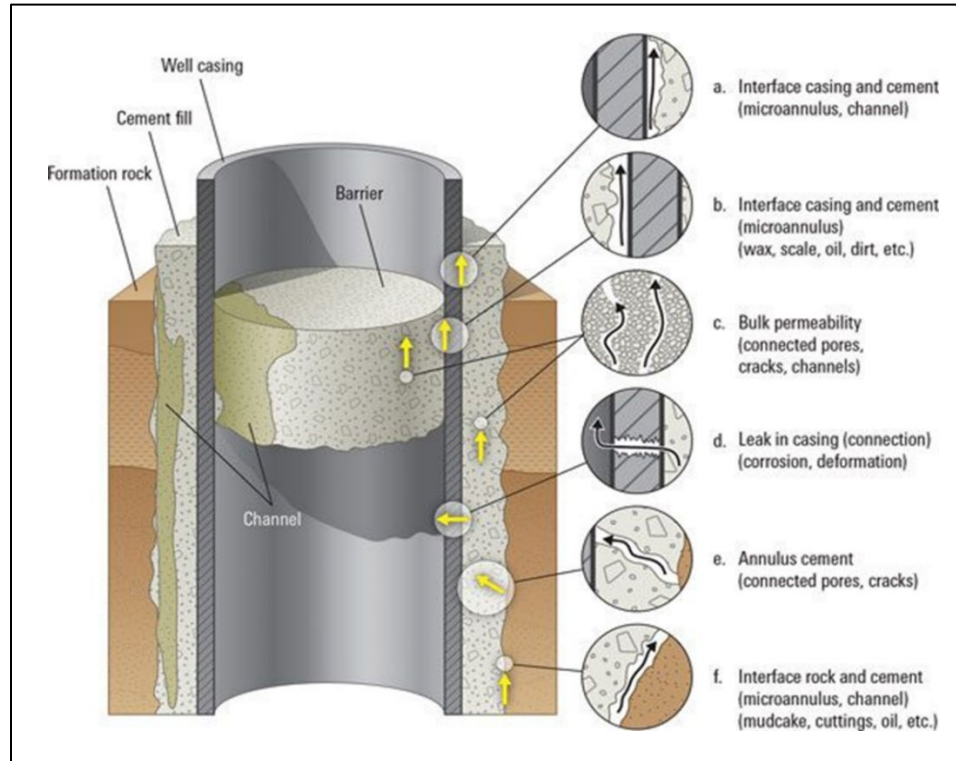


Figure 2.1. Cement Failures (Schlumberger, 2018)

## 2.2. CEMENT SEALANT MATERIALS

Typically, wells with poor primary cementing jobs or suspected leaks are repaired with cement squeeze operations, in which new cement is injected through perforations created in the casing near the suspected source of leakage to achieve proper zonal isolation. Squeeze cementing is a remedial process that involves the application of differential pressure across the cement slurry to accomplish the process of cement dehydration (Goodwin, 1984). In principle, the slurry is designed specifically to reach and fill the problem area, and create immobility until some compressive strength can be developed. However, cement slurry is often improperly placed or poorly designed due to the misjudgment of the leakage problem. The presence of an annular gap and/or fractures with

apertures on the order of 0.01–0.3 mm can still have a significant increase in effective permeability in the range of 0.1–1 mD (Um et al., 2014). Particularly, fractures or leakage pathways with small apertures are often difficult for conventional Portland cement to repair, as the cement slurry is potentially screened out from dispersing fluid and cannot enter the fracture. Therefore, squeeze cementing is often unsuccessful, and can result in a waste of rig time and high costs.

The main factor limiting the sealing performance of conventional Portland cement is the particle size. The commonly used Class H oil well cement contains large particles in the range of 100–150  $\mu\text{m}$  which makes it difficult to penetrate narrow channels, micro-annuli, or narrow mud channels and often lead to unsatisfactory results. In addition, bridging and cement dehydration will occur when the slurry is squeezed to penetrate fractures narrower than 400  $\mu\text{m}$ . Ultra-fine cement technologies, such as Halliburton's Micro Matrix cement and Schlumberger's SqueezeCRETE, have been developed to significantly reduce the particle size of the cement slurries. These new slurries have improved penetration capabilities through narrow slots.

Many researches have been conducted to study the rheology and factors affecting gel resistance to water flow. Grattoni et al. (2001) conducted a series of experimental work to link polymer gel properties (such as gel strength and polymer concentrations) to flow behavior. They found that permeability is a function of both water flow rate and polymer concentration. Yang et al. (2002) developed a mathematical model for the flow of water through channels impregnated with a polymer gel. Zhang and Bai (2010) showed that millimeter-sized particles formed a permeable gel pack in opening fractures rather than form full blocking.

Numerous studies have been conducted to evaluate in-situ gel propagation through fractures. Seright (1995, 1997, 1998, 1999, and 2001) studied both bulk gel placement and the mechanism behind gel propagation through fracture systems. Liu and Seright (2000) identified a correlation between gel rheology and extrusion properties of gels in fractures. Ganguly et al. (2001) conducted a series of experiments to determine the effects of fluid leak-off on gel strength when placed in fractures. Sydansk et al. (2005) characterized the transport of partially formed gels in fractures. Wang and Seright (2006) examined whether or not using rheology measurements to evaluate gel properties in fractures is an acceptable substitute for extrusion experiments as a way to reduce costs. Wilton and Asghari (2007) worked to determine how to improve bulk gel placement and performance through fractures. McCool et al. (2009) investigated the effect of shear on flow properties during the placement of sealants in fractures. . However, none of these previous work investigate the potential application of polymer gel to seal cracked and fractured cements.

Epoxy resin materials are being used to plug the cement pathways. This type of sealant seems to work effectively, but it needs more investigation to ensure proper placement and performance. Epoxy resin was first offered commercially in 1946 (Dewprashad & Eisenbraun, 1994). Epoxy is a product of the reaction between epichlorohydrin and bis-phenol A, and a common hardener is diethylenetriamine (Kabir, 2001). The viscosity of the epoxy resin can be altered using different types of diluents, such as ethylene glycol mono-butyl ether (Hakiki et al., 2015). The epoxy resin as a material offers many advantages over cement such as good viscosity. In addition, it is a solids-free material that can penetrate small gaps (Alsaihati et al., 2017; Todd et al., 2018), has good wetting and adhesive properties for mineral surfaces (Brooks et al., 1974),

especially silica surfaces (Shaughnessy et al., 1978), has good density and tunable setting time (Sanabria et al., 2016), and exceptional resistance to contamination (Perez et al., 2017).

In summary, this research will conduct an intensive experiment work to understand and evaluate the micro gel propagation and epoxy resin placement mechanisms in cement fracture and channel. The research will start by evaluate factors effect on sealant properties and rheology. Numerous experiments will then conduct to evaluate the micro gel injection through different size of fracture and channel. Several experiment will then conducted to evaluate water and oil leakages through gel and epoxy resin after placement.

## **PAPER**

### **I. THE POTENTIAL OF USING MICRO-SIZED CROSSLINKED POLYMER GEL TO REMEDIATE WATER LEAKAGE IN CEMENT SHEATHS**

#### **ABSTRACT**

Cementing is a major step in the construction and sealing of hydrocarbon wells. During the life cycle of the well, cement is prone to cracking due to a change in downhole conditions. This research investigates the use of micro-sized cross-linked polymer gel as a sealant material to mitigate cracked cement sheaths. Two experimental setups were designed to investigate water leakage through cement. The impact of polymer gel strength on the gel's ability to seal cement cracks was investigated using four gel strengths, including 500 pa, 1200 pa, 1450 pa, and 2440 pa. The impact of the width of the cement crack was also investigated using 0.5, 3.2, and 6.75 mm. Results showed that the polymer gel propagated across fractures like a piston with no gravity effect and with angle with gravity effect. Blocking efficiency to water flow is controllable, and it can be increased if a high strength polymer gel is selected. To the authors' knowledge, very little experimental work has been conducted to investigate the use of crosslinked micro-gel in cement zonal isolation. This study can provide the oil and gas industry with a better understanding of the materials to use in improving cement zonal isolation, and thus reduce the impact of cement failure.



## 1. INTRODUCTION

Wellbore integrity is defined by NORSOK D-010 (2013) as the “application of technical, operational and organizational solutions to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well.” If the cement is not properly completed and abandoned, it may develop leakages during any stage of the life of the well (Watson & Bachu, 2009). Fluids (water or hydrocarbon) can migrate through pathways within the cement itself or between the cement and its surroundings. When the wellbore integrity is compromised, these pathways occur and formation fluids are allowed to migrate between formations and/or from the formation to the surface. Leakage of water, gases, and hydrocarbon fluids through cement pathways may occur during the drilling or production stage of the well, or after the abandonment of the well, which may endanger personnel and the environment (Davies et al., 2014; Alkhamis & Imqam, 2018; Ahdaya & Imqam, 2019). The leakage of fluids along the interface between the wellbore and formation is a primary concern in hydrocarbon recovery (Dusseault et al., 2000). Leakage pathways in the cement annulus can happen due to mechanical failures as a result of pressure and temperature cycles, chemical degradation due to the corrosive formation fluids (Zhang & Bachu, 2010; and Liu et al., 2017), or due to improper slurry design. Improper slurry design includes the use of slurries with low densities, which may allow formation fluids to create channels within the cement sheath; the use of slurries with high fluid loss, which may affect the mechanical properties of the set cement; and the use of rigid cement, which can get fracked as a result of casing expansions and contractions. Also, failures can occur due to improper mud removal. The leftover mud on the surface of the formation and casing can prevent the

cement from bonding with its surroundings, creating micro-annuli. These and similar failures create pathways for formation fluids through the cement barrier.

Commonly, cement squeeze remedial operation is performed to seal cement leakages. However, cement squeeze cannot be used effectively to mitigate and prevent leakage because of low cement injectivity, pressure restriction, pin-hole leakage, micro-channels and fractures inside the leaking formation, and micro-cracks within primary cement (Jones et al. 2014). To overcome these limitations, self-healing cement was developed (Cavanagh et al. 2007; Roth et al. 2008; Reddy et al. 2010; and Khatri, 2013). Self-healing cement enables an automatic repair when micro-annuli, an internal cement crack, or another flow path is created. However, these kinds of sealing materials still have some problems associated with their swelling chemical additives and other concerns related to their mechanical and thermal stability. Crosslinked polyacrylamide polymer gels are another kind of sealant material that have been used in many oilfield applications due to their high injectivity and ability to mitigate excessive water migration; controlling the water production increases the oil recovery (Feng et al., 2018; Deng et al., 2018; and Sadati et al., 2019). Polymer gel could be activated by temperature, pressure, a change in the water salinity, and a change in the pH (Vasquez, J, and Santin, Y, 2015; Imqam et al., 2015), and the strength of the gel can be increased by implementing nanomaterials (Tongwa & Baojun, 2015).

Crosslinked polymer gels have also been widely known and used in the industry for conformance control, and the success of this material in enhancing hydrocarbon recovery is established (Seright, R, S, 1995; Bai. B et al., 2007; Seright, R S, 2009; Zhang, H., and Bai, B, 2010; Imqam, A et al., 2015; Yu et al., 2017; Imqam et al., 2018). Micro-

size particle gel is aggregated by water salinity. It forms at the surface, and then is dried and crushed into small particles to be injected into the reservoir. No gelation occurs in the reservoir (Imqam et al. 2015), though the gelation can be controlled by temperature and salinity (He et al., 2015; Zheng et al., 2017; and Fuseni et al., 2018). Yet, few laboratory studies investigate its ability in cement-zonal isolation. This study can provide the oil and gas industry with better understanding of the potential of using particle gel to mitigate the water leakage in cement sheaths. Thus, the ability of particle gels to plug the highly permeable zones behind casing or fractures or channels across the cement was the main motivation behind this work. The other motivation of this investigation is to test the injectivity of this material for zonal isolation application in comparing to the conventional cement materials.

This work investigates the potential of using micro-sized crosslinked polymer gel for water control to improve zonal isolation in cement sheaths through a set of measurements that count for the injectivity and the propagation of the gel particles inside the cement cracks or fractures. In addition, to the effect of the fracture sizes and the importance of defining the size of the cracks in the cement to increase the possibility of successful remedial job.

## **2. EXPERIMENTAL DESCRIPTION**

### **2.1. MATERIALS**

**Micro-Size Particle Gel.** A commercial super-absorbent polymer gel (Polyacrylamide-acrylic acid copolymer provided by Emerging Technologies Company) was selected as the sealant material for all experiments. The material was used as it received

from the emerging technologies company without any change in the chemical composition. Before swelling, the gel was a dry white granular powder.

Sodium Chloride (NaCl). A commercially available NaCl with a purity of 99.99% was used to create a brine solution.

Class H Cement. All the cement specimens that were used in this study were prepared using Class H cement and distilled water. The specific gravity of the cement was found to be 3.18, using a gas pycnometer. The chemical composition of Class-H cement was obtained using X-ray fluorescence spectroscopy (XRF) and is listed in Table 1.

Table 1. The chemical composition of class-H cement

<b>Comp.</b>	<b>CaO</b>	<b>SiO<sub>2</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>SO<sub>3</sub></b>
<b>Wt %</b>	65.72	20.36	6.19	3.17	2.26
<b>Comp.</b>	<b>MgO</b>	<b>K<sub>2</sub>O</b>	<b>SrO</b>	<b>TiO<sub>2</sub></b>	<b>Other</b>
<b>Wt %</b>	1.32	0.43	0.21	0.16	0.18

## 2.2. MATERIAL PREPARATION

Cement Paste Preparation. The cement slurry was mixed at room temperature using a two-speed bottom-drive blender. A specific amount of water was poured in the blender and then dry cement was added at a uniform rate while mixing at low speed for around 15 seconds. Then the blender was covered and the mixing continued for an extra 35 seconds at high speed (API RP 10B-2 2013). The cement slurry had a water/cement ratio (WCR) of 0.38 following the API specification 10A for API cement Class-H (API 2010).

Fractured Cement Cores Preparation. To prepare the fractured cement cores, one inch in diameter molds were cut into two halves (Figure 1 (c)). The cement was poured into each half and left for more than 72 hours to set. Then, two metal sheets with specific thickness (0.5 mm and 2.0 mm) were used to separate the two cement specimens to the targeted fracture width.

Gel Preparation. Initially, the gel particles were sieved using 20-40 mesh screens to obtain homogenous gel particles. Then, the brines with different concentrations were added to the gel particles and left to swell for 9-12 hours. After that, the gel particles were drained for 12 hours to remove excessive brine. For the gel plugging efficiency measurements, four swollen gel samples were prepared using four different NaCl concentrations (0.05, 0.25, 1, and 10%). The NaCl concentration ranges were selected based on the previous work that evaluated their impact on the swelling ratio and gel strength (Hao and Bai 2010; Tongwa and Bai 2015; Imqam et al 2015; Imqam et al 2018). For the gel propagation analysis, two different brine concentrations (0.05 and 1%) were prepared.

### **3. EXPERIMENTAL METHODOLOGY**

#### **3.1. RHEOLOGICAL MEASUREMENTS**

Anton Paar Modular Compact Rheometer MCR 302 Instruments with a parallel-plate system (PP50/TG using a 0.30 mm gap) were used to measure the gel strength. The measurements were conducted at 25 °C to measure the gel strength of the four swollen gel samples.

### 3.2. SWELLING GEL CAPACITY

Test tubes were used to immerse a specific amount of dry gel particles in a specific volume of brine solution. Four different brine solutions were used to determine the swelling capacity of the particle gel over time. The stable swelling ratio was computed for each concentration. The swelling ratios of the particle gel in different brine solutions were obtained using Equation (1) (Imqam et al., 2015):

$$\text{Swelling Capacity} = \frac{V_2 - V_1}{V_1} \quad (1)$$

where  $V_1$  is the initial volume of the gel sample before swelling, and  $V_2$  is the final volume of the gel sample after swelling.

### 3.3. EXPERIMENTAL SETUPS AND PROCEDURES

**3.3.1 Gel Propagation through Fracture.** The gel propagation through the cement setup is shown in Figure 1 (a). It is composed of a syringe pump, an accumulator with a piston, and an adjustable Plexiglass Cell. Gasket rubber was placed between the parallel plexi-glass plates, which mimics the fracture width. The transparent glass cell model was used to visualize and determine the gel flow mechanisms through the fracture. Cement usually has a very low water leakage into the matrix, and therefore the fracture cell setup was designed to mimic that and to only observe the flow performance along fractures. Gel and brine were injected through the accumulator into the plexiglass cell. Pressure transducers were placed at the inlet, in the middle, and at the outlet of the cell to observe the gel performance along the fracture. Three fracture widths (0.5 mm, 3.2 mm, and 6.75 mm) were used to examine the effect of the cement fracture size on the gel injection. The

fracture width sizes were selected along with gel swollen particle sizes to get different ratio ranges of gel particle size to fracture width. Table 2 shows the ratio of gel particle size to the fracture width for different gels. The ratios obtained were equal to one, below one, and above one to have a better understanding of gel passing mechanisms along different cement fracture width sizes.

Table 2. Characteristics of the Polymer Gel Particle/opening -Ratio Parameters

<b>Brine Concentration [% by weight]</b>	<b>Swollen Particle Gel Size [mm]</b>	<b>Fracture Width [mm]</b>	<b>Gel Particle Size to Fracture Width Ratio</b>
<b>0.05</b>	<b>4.88</b>	0.5	9.76
		3.2	1.525
		6.75	0.72
<b>1</b>	<b>3.2</b>	0.5	6.4
		3.2	1
		6.75	0.47

Gel Propagation through Fracture Procedure. Brine was initially injected into the Plexi-glass cell using an accumulator to mimic the case when the cement was already filled with

water before the sealant injection. After the brine was in place, particle gel was then injected into the Plexi-glass cell with a constant flow rate of 1 ml/min. The gel injection continued until the gel was produced from the outlet of the cell and the injection pressure became stable across the fracture. The injection pressure was recorded using the three pressure sensors, and a high accuracy camera was used also to monitor the angle of gel propagation across the fracture.

**3.3.2. Gel-Cement Plugging Efficiency Setup.** The gel-cement plugging efficiency setup, as shown in Figure 1 (b), is composed of a syringe pump, an accumulator with a piston, and a core holder. The pump is connected to the accumulator and is used to inject the brine and swollen gel. The cement fractured core as shown in Figure 1 (c) is placed in the core holder, and confining pressure is applied to prevent fluid leakage around the fractured core and to mimic the confining pressure conditions. Two fracture widths (0.5 mm and 2 mm) were used to examine the effect of cement fracture size on gel placement. A pressure transducer is used to record injection pressure.

**Gel-Cement Plugging Efficiency Procedure.** Gel was first injected into the fractured cement core through an accumulator with a constant flow rate of 1 ml/min. The injection stopped when the gel was produced from the core and gel injection stable pressure was achieved. After the gel was in place, brine was injected into the gel particle packed fracture to test the gel plugging efficiency to water. The brine was injected through an accumulator with a constant flow rate of 1 ml/min. The brine injection continued until the brine injection pressure became stable. The pressure data were recorded during all the experiments using transducers.



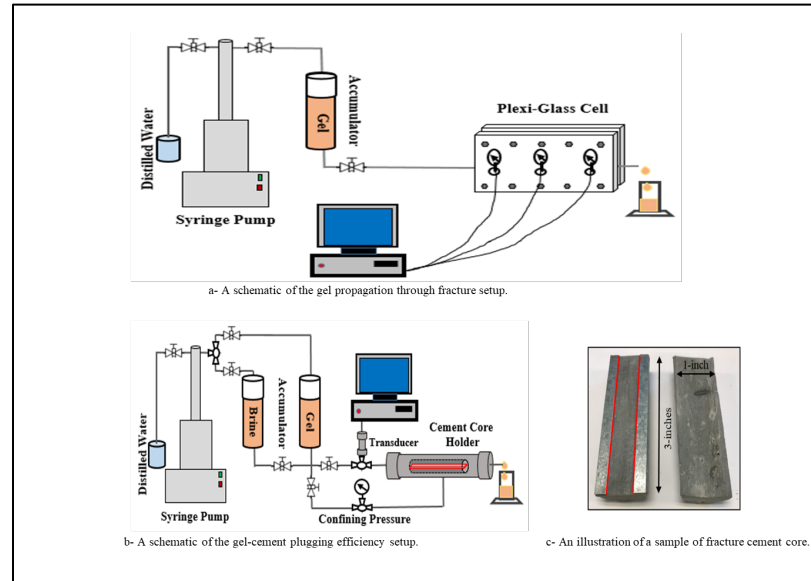


Figure 1. An illustration of the setups used in this study and a sample of fractured cement core

## 4. RESULTS AND ANALYSIS

The results for the rheology of the gel, the swelling capacity, gel propagation in the cement fracture, and gel plugging efficiency will be presented in this section.

### 4.1. POLYMER GEL SWELLING CAPACITY AND STRENGTH RESULTS

The ability of the particle gel to absorb water is a strong function of the (NaCl) concentration. As the brine concentration increases, the gel will absorb less water and its swelling will decrease. Lower gel swelling will result in a smaller particle size, but it will also result in stronger particles with higher gel strength. The change in brine concentration had a very clear effect on the swelling ratio of gel particles. The swelling ratio for the gel swollen in 0.05% NaCl reached 155 ml/ml, as shown in Figure 2 (A). The swelling ratio increased from 75 to 155 ml/ml when the brine concentration decreased from 0.25 % NaCl

to 0.05% NaCl. The gel particles that swelled more became weaker and began to soften. This decrease in gel strength is most likely due to the gel absorbing a large volume of water. Figure 2 (B) shows the measurement of the particle gel strength, which is represented by the storage modulus of the gel that was measured by the rheometer, swollen in different brine concentrations. The gel exhibited a significant increase in strength when the brine concentration increased.

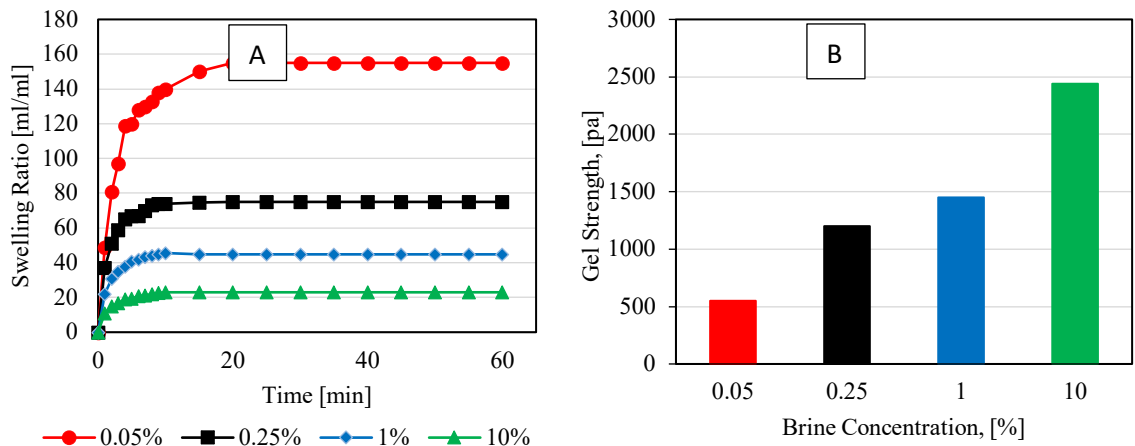


Figure 2. Swelling ratio and gel strength of gel at different NaCl (by weight) concentrations

## 4.2. GEL PROPAGATION THROUGH CEMENT'S FRACTURE

In this section, the movement of the gel inside the cement fractures was studied by utilizing the gel propagation through a transparent cement setup (Figure 1 (a)). This setup was intended to study the effects of gel particle size to fracture width ratio on gel propagation performance, as described in Table 2.

**4.2.1. Gel Flow with no Gravity Effect.** Figure 3 is divided into two sets of experiment observations, I and II: Set (I) shows the gel particles' movement inside the

model for fracture of width 0.5 mm and gel strength of 550 pa. Set (II) shows the gel particles' movement inside the model for the same fracture width but for gel strength of 1450 pa. These two sets of visual results were for the gel size/fracture width ratio larger than one, in which the gel particle size was greater than the fracture width. The gel particles flow was monitored at the fracture inlet (Figure 3 (A)) through the fracture center (Figure 3 (B)) and at the fracture outlet (Figure 3 (C)) using digital camera video. Piston-like gel movement (no signs of gravity effect) across the fracture was observed for the two sets of experiments as it is shown by the colored light blue dot lines. Figure 4 shows the gel transport angle measurements along the fracture length. It is observed that gel flow inside the fracture started with a small angle (40 degrees) at the fracture inlet. However, as the gel transported deep into the fracture, the flow behavior became a piston-like (90 degrees). This result partially agrees with the finding of (Zhang, & Bai, 2010), where they observed that piston-like occurred when the swollen particle sizes were close to the width of the fracture.

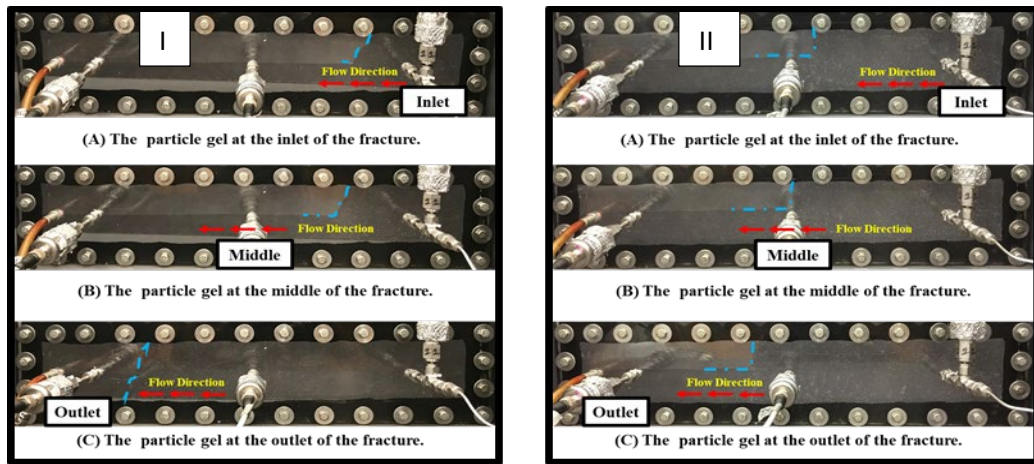


Figure 3. Gel propagation through fractures: (I) gel particle size to fracture width ratio is 9.76, while (II) particle size to fracture width ratio 6.4

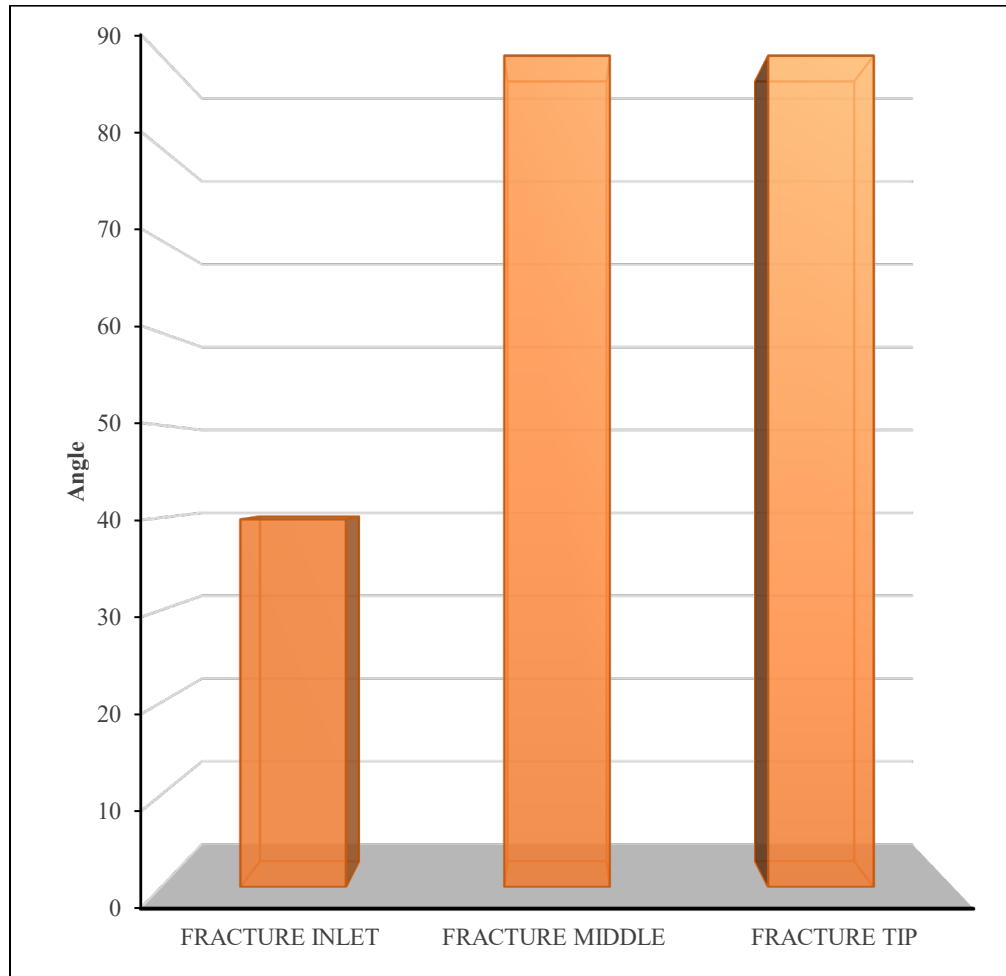


Figure 4. Gel propagation angles (gel particle size to fracture width ratio is 6.4)

Figure 5 (A) shows injection pressure distribution across the fracture for the gel strength of 1450 pa. An increase in pressure was noticed during particle gel movement and stabilized at inlet fracture (46 psi), middle fracture (33 psi) and outlet fracture (28 psi). Interestingly, results in Figure 5 (B) showed that when the ratio increased from 6.4 to 9.76 (using a gel strength of 550 pa), gel injection pressure decreased instead of increasing. This indicates that not always the gel particle size to the fracture width is the main controller of the passing mechanism but also the gel strength. The gel strength samples of 550 pa had a larger size than the gel strength samples of 1450 yet it has less injection pressure.

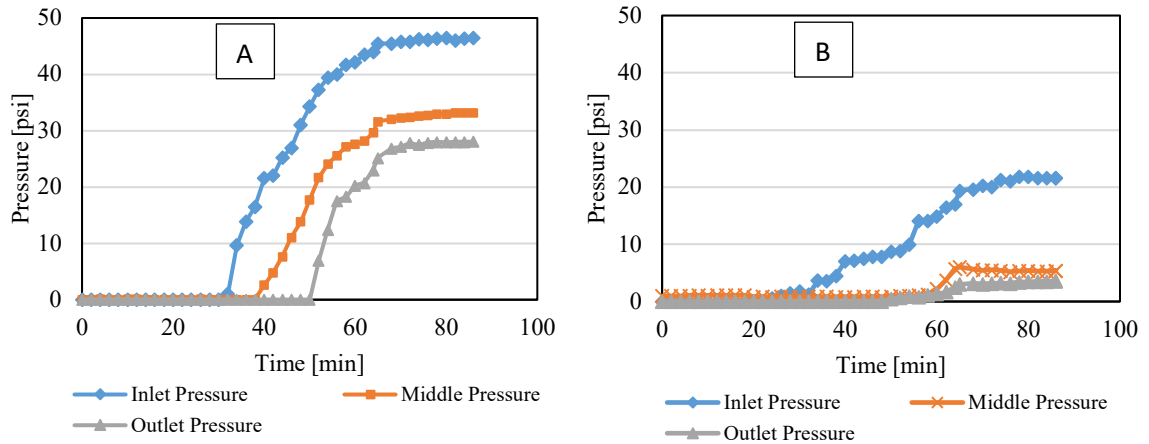


Figure 5. (A) Gel propagation injection pressure through fractures width of 0.5 mm using 1450 pa strength (gel size/fracture width ratio is 6.4) and (B) Gel propagation through fractures width of 0.5 mm using 550 pa strength (gel size/fracture width ratio is 9.76)

**4.2.2. Gel Flow with Gravity Effect.** Figure 6 I and II shows visual gel propagation performance through fractures. Set I is for gel samples of strength 550 pa transported along a fracture width of 3.2 mm. Set II is for gel samples of strength 550 pa transported along fracture width of 6.75 mm. Similarly, Figure 7 I and II shows visual observations of gel samples of strength 1450 pa transported along a fracture width of 3.2 mm and 6.75 mm, respectively. These four sets of experiments were for the gel size/fracture width ratio equal to or below one, where the gel particle sizes were equal to or smaller than fracture width sizes. All the observations for gel particles move along the fracture as indicated by the colored light blue dot lines showed that the gel propagated with an inclination or steeper inclination performance along the fracture length. As a result, the gel particles were observed to settle down into the fracture due to the gravity effect. Figure 8 shows the gel transport angle measurements along the fracture length. At the fracture inlet, gel particles transported with 25 degrees, whereas at the fractured middle the angle became 32 degrees,

which is steeper, and finally, at the fracture tip, the angle became 45 degrees. This indicates that as the gel particles move along the fracture, it will begin to develop a steeper angle and thus will not fill the fracture completely, which may affect its ability for water leakage remediation.

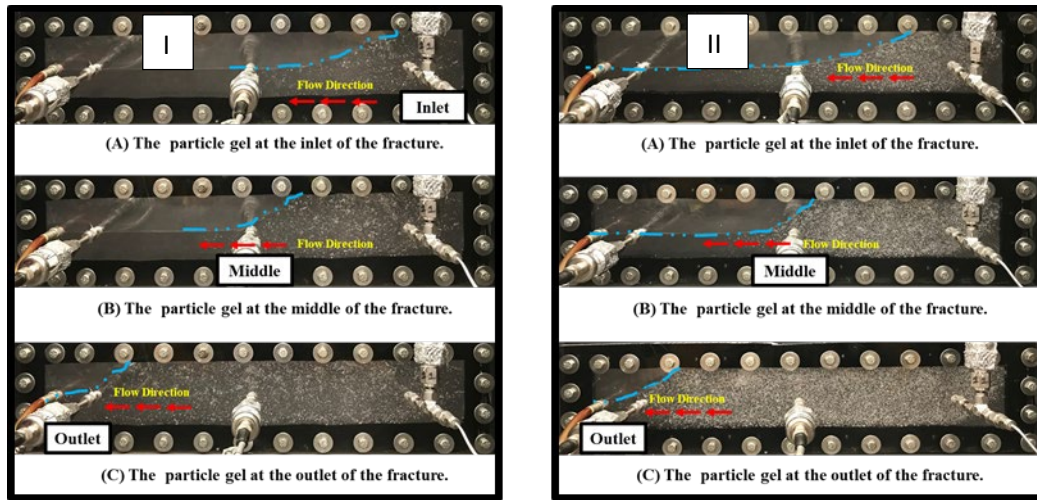


Figure 6. Gel propagation through fractures: (I) gel particle size fracture width ratio is 1.5, (II) gel particle size fracture width ratio is 0.7

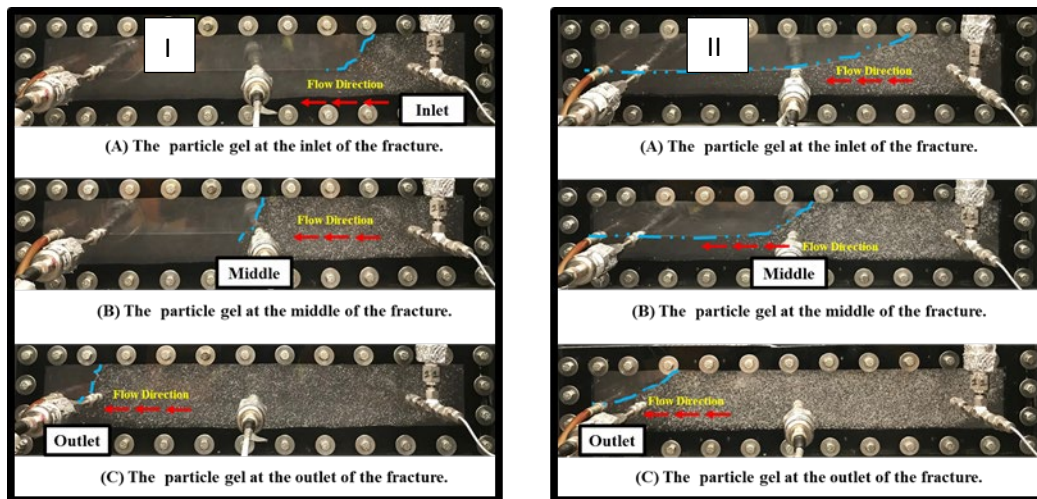


Figure 7. Gel propagation through fractures: (I) gel particle size fracture width ratio is 1.0, (II) gel particle size fracture width ratio is 0.47

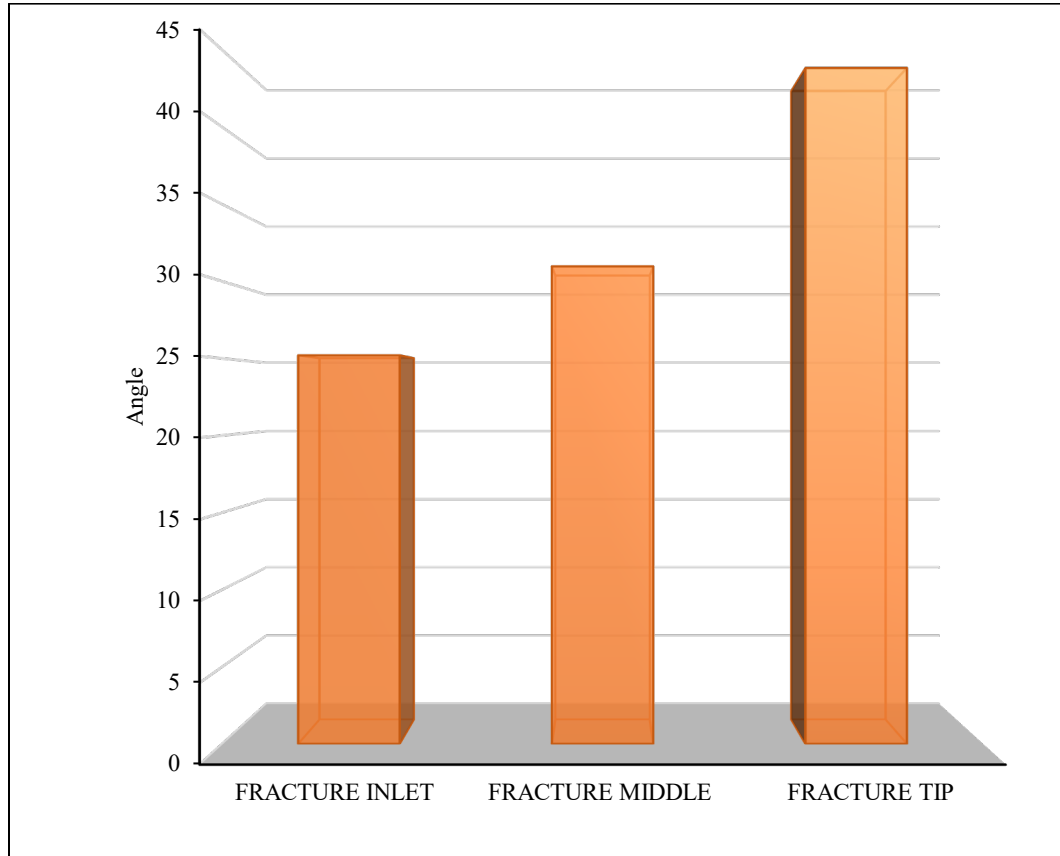


Figure 8. Gel propagation angles (gel particle size fracture width ratio is 0.47)

Figure 9 shows the pressure change along the fracture until stabilization. Referring to the pressure distribution measurements in Figure 5 (A) and (B), gel injection pressure presented in Figure 9 was much lower, and this occurred because the gel particle size to fracture width ratio was smaller than 1. The stable pressure was 12.4 psi at the fractured inlet, 8.3 psi at the fractured middle, and 3.8 psi at the fractured outlet. This also shows that as the gel moves further into the fracture, the pressure started to decrease, which is an indication that high gel injection pressure may be required for larger and deeper fractures or cracks. Also, since the pressure at the fracture tip was lower compared to the fracture inlet, the gel may have a lower plugging efficiency at the fracture tip compared to the fracture inlet.

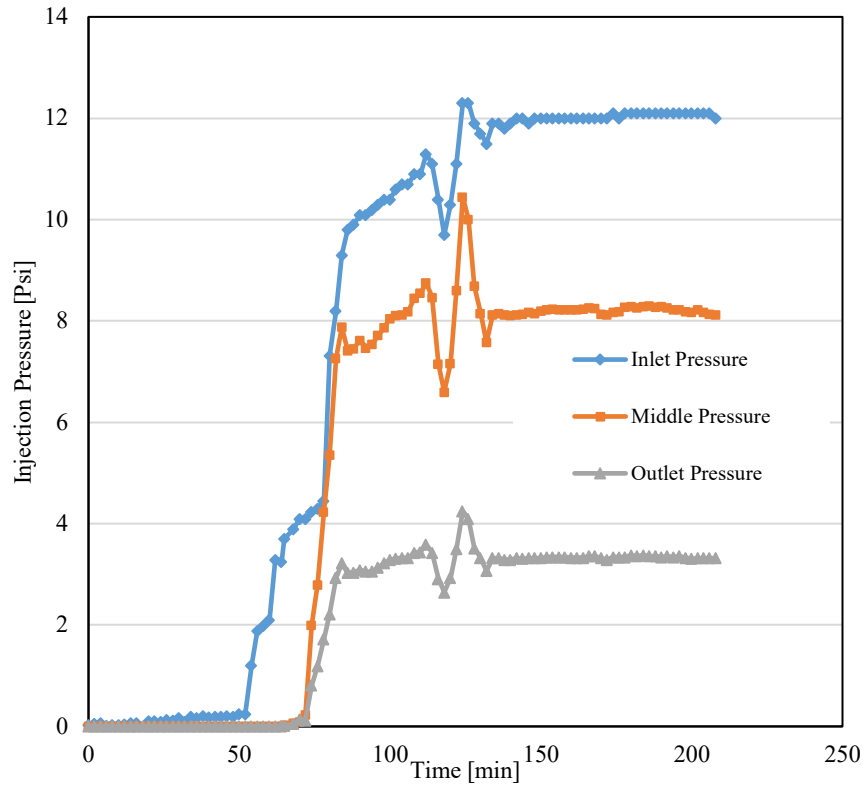


Figure 9. Gel propagation injection pressure through fractures 6.75 mm using 1450 pa strength (gel particle size fracture width ratio is 0.47)

**4.2.3 Gel Particles Flow Mechanisms.** Table 3 shows the summary of gel transport mechanisms of using different gel strengths (550 pa and 1450 pa), two sizes of particle gel (4.88 mm and 3.2 mm), and three fracture widths (0.5 mm, 3.2 mm, and 6.75 mm). The gel movement was piston-like when  $(D_g/W_f)$  was much larger than 1. However, gel movement with angle occurred when  $(D_g/W_f)$  was close to or smaller than 1. Also, the flow direction changed from inclined to a steeper inclined as the  $D_g/W_f$  became smaller than 1. The piston-like angle started with an angle less than  $90^\circ$  at the fracture inlet and increased to  $90^\circ$  as the gel particle moved deep into the fracture. However, when  $D_g/W_f$  smaller than or equal to 1, the angle started with less than  $90^\circ$  and kept decreasing below  $90^\circ$  as the gel moved deep into fracture due to the gravity effect.



Table 3. A summary of the gel particle flow mechanisms across fractures or cracks

Gel Strength [pa]	Particle Gel Size, Dg [mm]	Fracture Width, Wf [mm]	Dg/Wf	Gel Flow Mechanism	
				Mechanism	Angle [degree]
550	4.88	0.5	9.76	Piston-like	30 -90
		3.2	1.53	Inclined	20 - 25
		6.75	0.72	Steeper inclined	20 – 38
1450	3	0.5	6	Piston-like	40 -90
		3.2	0.93	Inclined	40 -70
		6.75	0.44	Steeper inclined	25 - 45

#### 4.3. GEL-CEMENT PLUGGING EFFICIENCY

In this section, the ability of the gel to plug cement fractures and control water production was tested using the Gel-Cement Plugging Efficiency setup (Figure 1b).

**4.3.1. Effect of Gel Strength.** Figure 10 shows gel injection pressure for different gel strengths. A cement fracture with 0.5 mm was used in this investigation. Gel injection pressure increases as the gel strength increases. The highest gel strength 2440 pa provided the highest pressure value of 70 psi. When the gel strength decreased to 1450, 1200, 550 pa, the injection stable pressure decreased to 55, 48, and 40 psi, respectively. These results indicate that gel injectivity can be controlled by designing different gel strengths. In referring to the literature, these results show gel particle operation has better injectivity than squeeze cementing jobs. This occurred because gel particles are elastic and deformable materials which allow for this high injectivity compared to solid cement materials.

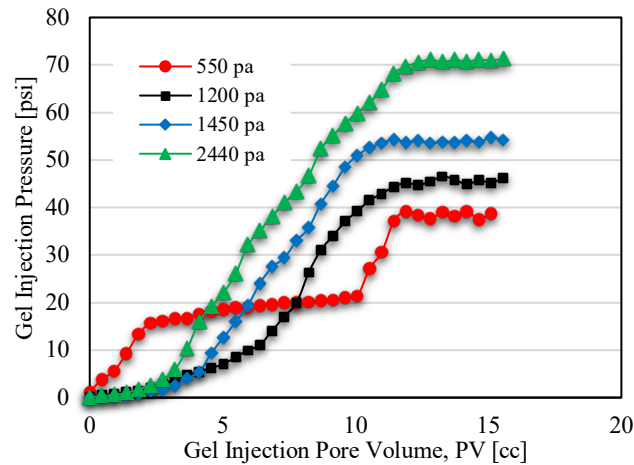


Figure 10. Particle gel injection (0.5 mm Fracture width)

**4.3.2. Resistance to Water Flow after Gel Placement.** Water Breakthrough Pressure Measurements. After gel placement within the cement fracture, brine was injected with constant flowrate to characterize the particle gel blocking behavior against water. Figure 11 (A) shows that the gel with the highest gel strength resulted in the highest pressure to reach a water breakthrough. Gel with 2240 pa strength had the highest water breakthrough pressure at 70 psi, and it decreased to approximately 35 psi when the gel strength decreased to 550 pa. After the water breakthrough, the pressure decreased until it reached stable pressure. Stronger gel still had higher brine stable pressure compared to gels with lower strengths. The ability of the gel to plug the cement fractures and manage water production is also controllable, and this ability increased by increasing the gel strength.

Water Residual Resistance Factor Calculations (Frrw). It is defined as the ratio of water phase permeability before and after particle gel treatment. As the Frrw value increases, the gel's plugging efficiency increases, and thus a high Frrw value is favorable. Equation 2 shows how Frrw was calculated in this research.

$$Frrw = \frac{Brine\ Stable\ Pressure_{After\ Gel}}{Brine\ Stable\ Pressure_{Before\ Gel}} \quad (2)$$

Figure 11 (B) shows that by increasing gel strength, Frrw increases. This indicates that increasing gel strength is more effective for water shut-off applications. Frrw increased as the gel strength increased; for instance, the Frrw of 550 pa gel was 34.5, while the Frrw of 2440 pa gel was 12136. The gel blocking efficiency (E) can be represented as the percentage of permeability reduction, which can be calculated from Equation 3 (Imqam et al. 2015).

$$E (\%) = \left[ 1 - \left( \frac{1}{Frrw} \right) \right] * 100 \quad (3)$$

The results showed that the plugging efficiency increased when a strong gel was selected as a sealant agent, and Frrw ranges from 97-99% for all gel strengths. Table 4 lists a summary of gel strength, Frrw, and blocking efficiency.

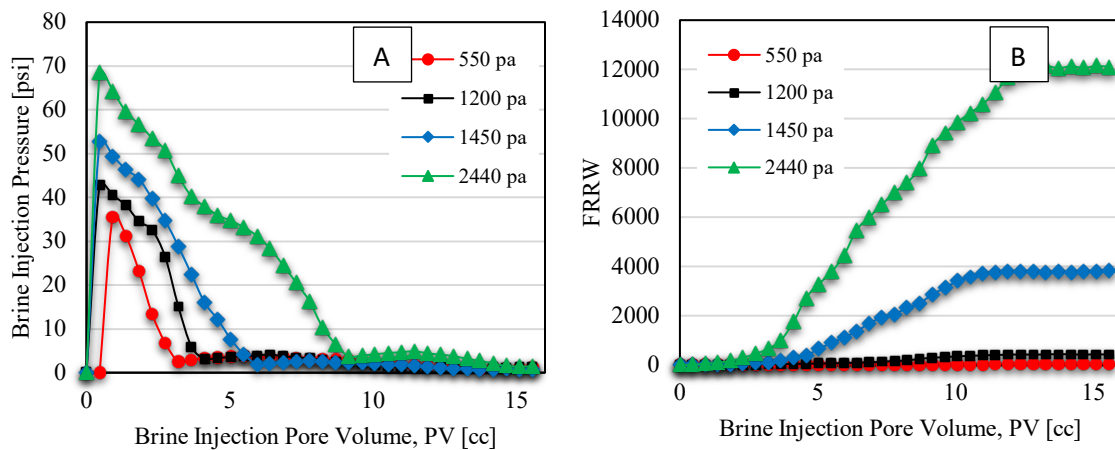


Figure 11. (A) Brine injection pressure and (B) the Frrw (0.5mm Fracture width)

In all experiments, it has been noticed that the brine injection pressure increased sharply until reaching a certain peak, at which point it began to decline. This peak indicates the point at which gel failure and washout began to occur in each section (Seright, 2003). After each peak, the pressure declined significantly before becoming stable in all sections. Also, pressure decline was noticed to decline with two humps behavior or single hump behavior based on the gel strength. The hump pressure behavior could occur again if high gel strength was used where it is more resistance to water flow occurs compared to gels with low strength.

Table 4. A summary of gel strength, Frrw, and blocking efficiency

<b>Gel Strength [Pa]</b>	550	1200	1450	2440
<b>Frrw [fraction]</b>	34.5	425	3838	12136
<b>Blocking Efficiency [%]</b>	97.1	99.77	99.97	99.99

**4.3.3. Effect of Cement's Fracture Width.** Cement can fail in the annuli of the wellbore for several reasons, and the fractures that are created can have different shapes and sizes. The performance of the gel as a sealant can be affected by the geometry of the fractures. Thus, it is essential to study the effects of the size of the fractures on the sealant before applying any remedial job. In this section, a comparison and analysis are presented to point out the effect of cement fracture width on the gel plugging efficiency. Gel with strength of 1450 pa was used for this investigation. The results of particle gel injection in a fracture of 2 mm width showed a trend similar to that of 0.5 mm width, as shown in

Figure 12. However, increasing the fracture width from (0.5 mm) to (2.0 mm) decreased the gels stabilized pressure from approximately 55 psi to 40 psi, respectively.

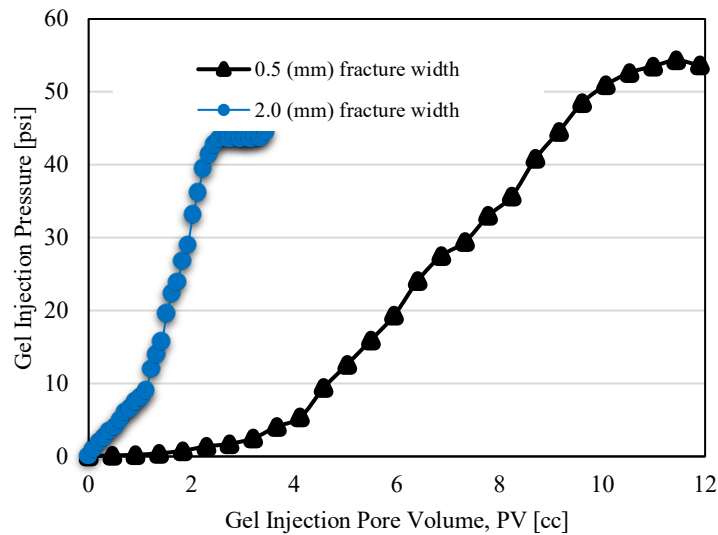


Figure 12. Particle gel injection in 0.5 mm and 2.0 mm fracture width using 1450 pa gel strength

**4.3.4. Resistance to Water Flow after Gel Placement.** Water Breakthrough Pressure Measurements. The water breakthrough pressure decreased as the fracture width increased at the same injection flowrate, which can be seen in Figure 13 (A). The water breakthrough pressure was around 53 psi for the 0.5 mm fracture width and dropped to almost 43 psi for the 2.0 mm fracture width. By increasing the fracture width, the ability of the gel to plug the cement fracture decreased. Figure 13 (B) provides information about the effect of the fracture width on the Frrw. The gel strength is 1450 pa and the fracture widths are 0.5 mm and 2 mm. Frrw increased when increasing the fracture width. Table 5 lists the summary of Frrw and blocking efficiencies for 1450 pa gel strength. The blocking efficiencies were 97% and 99% for 0.5 mm and 2.0 mm fracture widths, respectively.

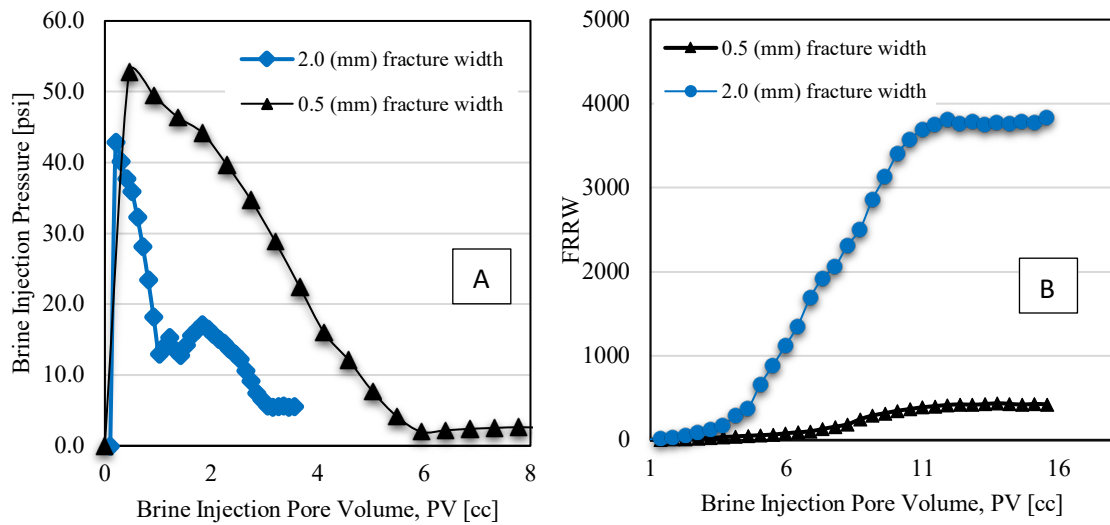


Figure 13. (A) Brine injection in 0.5 mm and 2.0 mm fracture width using 1450 pa gel strength and (B) Frrw in 0.5 and 2.0 mm fracture width using 1450 pa gel strength

Table - 5 Summary of Frrw and Blocking Efficiency for (1450 pa) gel strength

Fracture Width [mm]	0.5	2
Frrw [fraction]	425	3838
Blocking Efficiency [%]	97	99.9

## 5. CONCLUSIONS

By studying the effects of injecting particle gel in cement fractures, several findings were obtained in this study. These findings are based on the analysis of gel swelling capacity measurements, gel strength, gel transportation tests, and gel plugging efficiency experiments. The main conclusions are summarized below:

- Polymer gel particles show an acceptable injectivity performance through smaller fractures widths and crack features. However, their plugging performance to water leakage is limited to less than 100 psi. This failure pressure could be controlled by managing the gel strength, but to a certain level.
- The gel particles propagated piston-like when the gel particle size to fracture width ratio was larger than 1. However, it propagates with different angles when the gel particle size to fracture width ratio was equal to or below 1. Thus, it is essential to consider the effect of the cement fracture widths or cracks gaps when gel particles are considered for remedial operations.
- Selecting the gel sealant material not only depends on the plugging efficiency but also on the gel injectivity performance. The gel with high strength had better blocking efficiency but lower injectivity when compared with gel has low strength, and vice versa.

### **ACKNOWLEDGMENT**

The author wishes to thank King Abdulaziz University (KAU) for granting him his scholarship.

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## **II. THE APPLICATION OF MICRO-SIZED CROSSLINKED POLYMER GEL FOR OIL AND WATER CONTROL IN CHanneled CEMENT CORES**

### **ABSTRACT**

Cementing is a major step in the construction and sealing of hydrocarbon wells. During the life cycle of a well, cement is prone to cracking due to change in downhole conditions. This research investigates the use of micro-sized cross-linked polymer gel as a sealant material to mitigate cracked cement sheaths. An experimental setup was designed to investigate fluids leakage through channels in the cement. The impact of the size of the cement channel was investigated using 1.15 and 1.5 mm diameter. Results show that the blocking efficiency of the polymer gel to fluid flow is controllable. To the authors' knowledge, very little experimental work has been conducted to investigate the use of crosslinked micro-gel in cement zonal isolation. This study provides the oil and gas industry with an innovative method to improve cement zonal isolation, thus reducing the impact of cement failure.

### **1. INTRODUCTION**

Wellbore integrity is defined by NORSOK D-010 (2013) as the “Application of technical, operational and organizational solutions to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well.” If the cement is not properly completed and abandoned, it may develop leakages during any stage of the life of the well (Watson and Bachu, 2009). Fluids (water or hydrocarbon) can migrate through pathways

within the cement itself or between the cement and its surroundings. When the wellbore integrity is compromised, these pathways occur and formation fluids can migrate between formations and/or from the formation to the surface. Leakage of water, gases, and hydrocarbon fluids through cement pathways may occur during the drilling stage of the well, the production stage, or after the abandonment of the well, which may endanger personnel and the environment (Davies et al., 2014; Alkhamis, M. and Imqam, A., 2018; Ahdaya, M. and Imqam, A., 2019). The leakage of fluids along the interface between the wellbore and the formation is a primary concern in hydrocarbon recovery (Dusseault et al., 2000). Leakage pathways in the cement annulus can occur due to mechanical failures caused by pressure and temperature cycles, chemical degradation due to the corrosive formation fluids (Zhang and Bachu, 2010), or improper slurry design. Improper slurry design includes the use of slurries with low densities, which may allow formation fluids to create channels within the cement sheath, the use of slurries with high fluid loss, which may affect the mechanical properties of the set cement, and the use of rigid cement, which can be fracked as a result of casing expansions and contractions. In addition, failures can occur due to improper mud removal. The leftover mud on the surface of the formation and casing can prevent the cement from bonding with its surroundings, creating micro-annuli. These failures and more can happen creating pathways for formation fluids to flow through the cement barrier.

Commonly, a cement squeeze remedial operation is performed to seal cement leakages. However, cement squeeze in certain situations cannot be used effectively to mitigate and prevent leakage because of the cement's low injectivity, pressure restriction, pin-hole leakage, channels and fractures inside the cement, and micro-cracks within the

primary cement (Jones et al., 2014). To overcome these limitations, self-healing cement was developed (Cavanagh et al., 2007; Roth et al., 2008; Reddy et al., 2010; Khatri. 2013). Self-healing cement automatically repairs itself when micro annuli, internal cement cracks, or other flow paths are formed. However, self-healing cement still has some problems associated with swelling chemical additives and other concerns related to mechanical and thermal stability. Crosslinked polyacrylamide polymer gel are another kind of sealant material that have been used in many oilfield applications due to their high injectivity and ability to mitigate hydrocarbons and excessive water migration. Polymer gel could be activated by temperature, pressure, change in the water salinity, and change in the pH (Vasquez, J. and Santin, Y., 2015; and Imqam et al., 2015). Crosslinked polymer gels have been widely known and used in the industry for conformance control and the success of this material in enhancing hydrocarbon recovery is established due to its ability significantly to reduce water production with minimal impact on oil production (Seright, R, S, 1995; Bai. B et al., 2007; Seright, 2009; Zhang, H., and Bai, B, 2010; Imqam, A. et al., 2015; Liang, B. et al., 2017 and Imqam et al., 2018; Al-Saedi et al., 2018). Micro-size particle gel is developed at the surface and then dried and crushed into small particles to be injected into the reservoir. No gelation occurs in reservoir (Imqam et al., 2015). The ability of particle gels to plug the high permeable zones of reservoirs in order to enhance water injection recovery was the main motivation behind investigating the ability of the same material to plug the leakages of cement near producing zones. The other motivation of this investigation is the low injectivity of this material if used for zonal isolation when compared to conventional cement (Vasques, J and Santin, Y 2015).

This work investigates the potential of using micro-sized crosslinked polymer gel for fluid control to improve zonal isolation in the cement sheath through a set of measurements that accounts for the injectivity of the gel particles inside the cement channels, in addition, to the effect of the channel sizes and the importance of defining the size of the channels in the cement to increase the possibility of a successful remedial job.

## **2. EXPERIMENTAL DESCRIPTION**

### **2.1. MATERIALS**

**Micro-Size Particle Gel.** A commercial superabsorbent polymer gel was selected as the sealant material for all experiments. The main component of the gel is a crosslinked polyacrylic acid/polyacrylamide copolymer. Before swelling, the gel was a dry white granular powder.

**Sodium Chloride (NaCl).** Commercially available NaCl with a purity of 99.99% was used to create the brine solution.

**Class H Cement.** All of the cement specimens that were used in this study were prepared using American Petroleum Institute (API) Class H cement and distilled water. The specific gravity of the cement was found to be 3.18 using a gas pycnometer. The chemical composition of Class-H cement was obtained using X-ray fluorescence spectroscopy (XRF) and is listed in Table 1.

Table 1. The chemical composition of class-H cement

<b>Comp.</b>	<b>CaO</b>	<b>SiO<sub>2</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>SO<sub>3</sub></b>
<b>Wt %</b>	65.72	20.36	6.19	3.17	2.26
<b>Comp.</b>	<b>MgO</b>	<b>K<sub>2</sub>O</b>	<b>SrO</b>	<b>TiO<sub>2</sub></b>	<b>Other</b>
<b>Wt %</b>	1.32	0.43	0.21	0.16	0.18

## 2.2. MATERIALS PREPARATION

**Cement Paste Preparation.** The cement slurry was mixed at room temperature using a two speed bottom-drive blender. A specific amount of water was poured in to the blender, and then dry cement was added at a uniform rate while mixing at low speed for around 15 seconds, after which the blender was covered and mixing continued for extra 35 seconds at high speed (API RP 10B-2 2013). The cement slurry had a water/cement ratio (WCR) of 0.38 in accordance to API specification 10A for API cement Class-H (API, 2010).

**Channeled Cement Core Preparation.** In preparation, cement cores with channels, were filled with cement. Wires of 1.15 and 1.5 mm in diameter were placed in 1 in, diameter molds to create artificial channels in the cement cores. The cement was poured into the molds and left for more than 72 hours to set. Some of the wires were placed uniformly, and some wires were placed non-uniformly to observe the difference between the two cases. Figure 1 illustrates the channeled cement core.

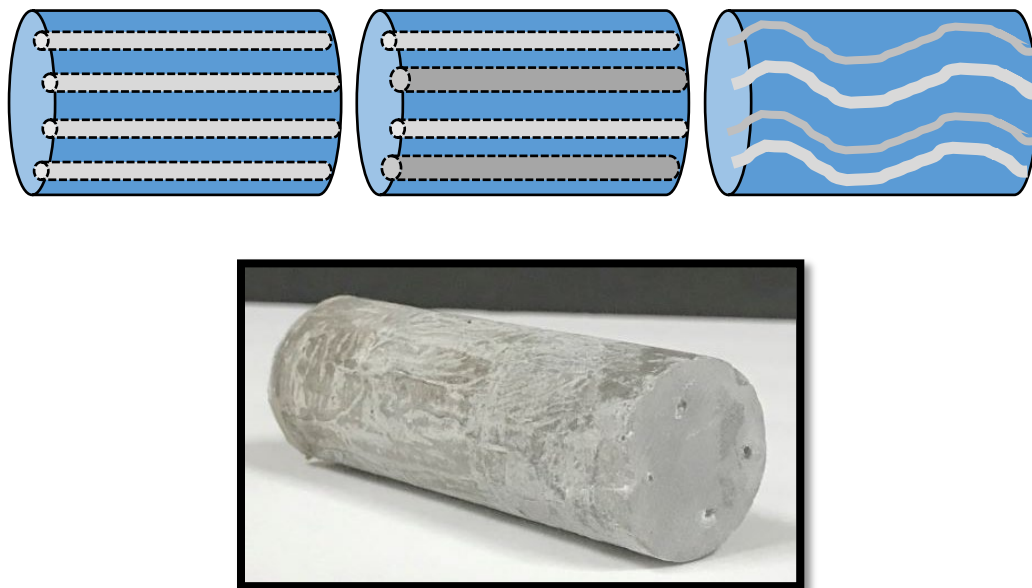


Figure 1. An illustration of channeled cement core

Gel Preparation. Initially, the gel particles were sieved using 20–40 mesh screens to obtain homogenous gel particles. Then, a 1% concentration brine was added to the gel particles, which were left to swell for 9–12 hours. After this, the gel particles were drained for 12 hours to remove excess brine.

### 3. EXPERIMENTAL METHODOLOGY

#### 3.1. RHEOLOGICAL MEASUREMENTS

An Anton Paar Modular compact rheometer (MCR) 302 with a parallel-plate system (PP50/TG using a 0.30 mm gap) was used to measure the gel strength. The measurements were conducted at 25°C. A gel sample was prepared using a 1% brine concentration following the gel preparation procedure mentioned previously.



### 3.2. PLUGGING EFFICIENCY

Oil Residual Resistance Factor Calculations ( $F_{rro}$ ). This is defined as the ratio of the water phase permeability before and after particle gel treatment. As the  $F_{rro}$  value increases, the gel's plugging efficiency increases, and thus a high  $F_{rro}$  value is favorable. Equation 1 shows how  $F_{rro}$  was calculated in this research.

$$F_{rro} = \frac{Oil\ Stable\ Pressure_{After\ Gel}}{Oil\ Stable\ Pressure_{Before\ Gel}} \quad (1)$$

Water Residual Resistance Factor Calculations ( $F_{rrw}$ ). It is defined as the ratio of water phase permeability before and after particle gel treatment. As the  $F_{rrw}$  value increases, the gel's plugging efficiency increases, and thus a high  $F_{rrw}$  value is favorable. Equation 2 shows how  $F_{rrw}$  was calculated in this research:

$$F_{rrw} = \frac{Brine\ Stable\ Pressure_{After\ Gel}}{Brine\ Stable\ Pressure_{Before\ Gel}}. \quad (2)$$

Gel-Blocking Efficiency Calculations ( $E$ ). The gel-blocking efficiency can be represented as the percentage of permeability reduction, which can be calculated from Equation 3 (Imqam et al., 2015):

$$E\ (\%) = \left[ 1 - \left( \frac{1}{F_{rr}} \right) \right] * 100. \quad (3)$$

Gel-Cement Plugging Efficiency Setup. The gel-cement plugging efficiency setup, as shown in Figure 2, is composed of a syringe pump and an accumulator with a piston and a core holder. The pump is connected to the accumulator and is used to inject the brine and swollen gel. The accumulator has a piston inside to prevent the fluid from contacting the distilled water injected through the pump. The cement channeled core (see Figure 2) is

placed in the core holder, and confining pressure is applied to prevent fluid leakage around the channeled core and to mimic the formation pressure. Three cement cores with different dimensions were used to examine the effect of cement's channel size on the gel placement. A pressure transducer was used to record the injection pressures.

**Gel-Cement Plugging Efficiency Procedure.** Brine or oil was first injected into the channeled cement core through an accumulator with a constant flowrate of 2 ml/min to determine the injectivity of the channeled cores. Then, the gel was injected in the same manner. The injection stopped when the gel was produced from the core and stable gel injection pressure was achieved. After the gel was in place, brine or oil was injected in the gel particle packed channels to test the gel plugging efficiency to those fluids. The injection flow rate was 2 ml/min.

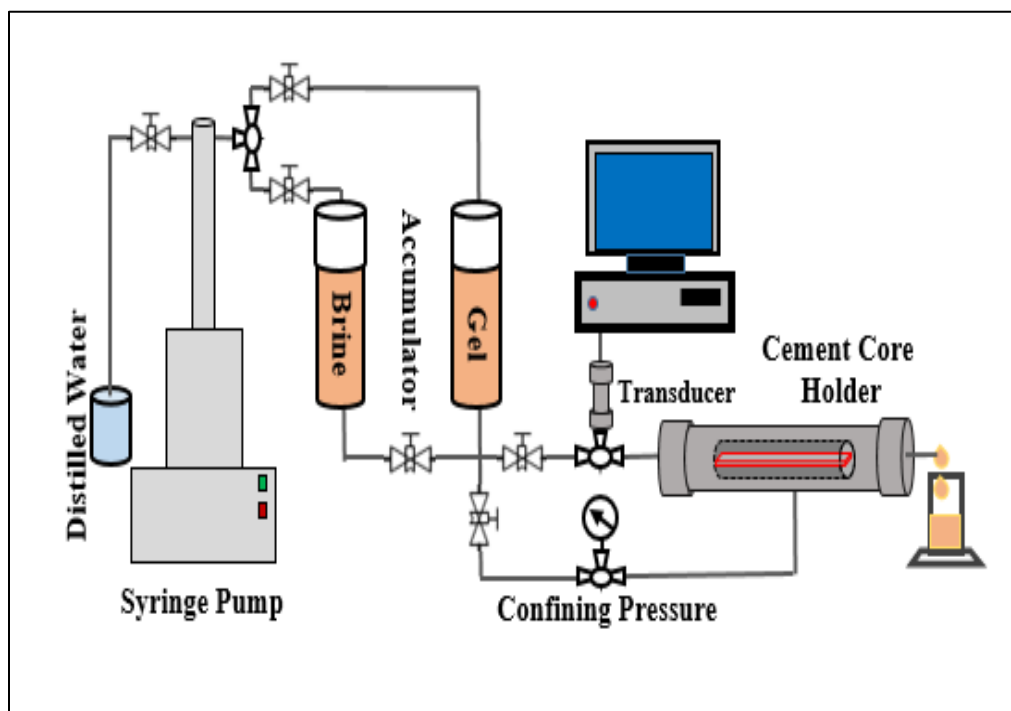


Figure 2. A schematic of the gel-cement plugging efficiency setup

## 4. RESULTS AND ANALYSIS

The results for the rheology of the gel and gel-plugging efficiency will be presented in this section.

### 4.1. RHEOLOGICAL MEASUREMENTS RESULTS

The ability of the particle gel to absorb water largely depends on the NaCl concentration. As brine concentration increases the gel will absorb less water and its swelling will decrease. Lower gel swelling will result in smaller particles size, but stronger particles with higher gel strength. The change in brine concentration had a very clear effect on the swelling ratio of gel particles. The swelling ratio for the gel swollen in 0.05% brine reached 155 ml/ml as shown in Figure 3. The swelling ratio increased from 75 to 155 ml/ml when the brine concentration decreased from 0.25 to 0.05%. The gel particles that swelled more, became weaker, and began to soften. This decrease in gel strength is most probably due to the gel absorbing a large volume of water. The concentration of NaCl had a strong impact on the water absorption capacity.

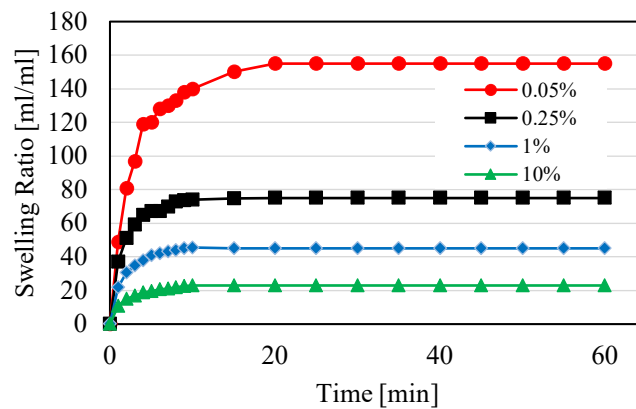


Figure 3. Swelling ratio of gel at different NaCl concentrations

Figure 4 shows the measurement of the particle gel strength, which is represented by the storage modulus of the gel, measured by a rheometer, swollen in different brine concentrations. The gel exhibited a significant increase in strength when the brine concentration increased. The swelling ratio was restricted (see Figure 3), which caused an increase in gel strength in higher brine concentrations.

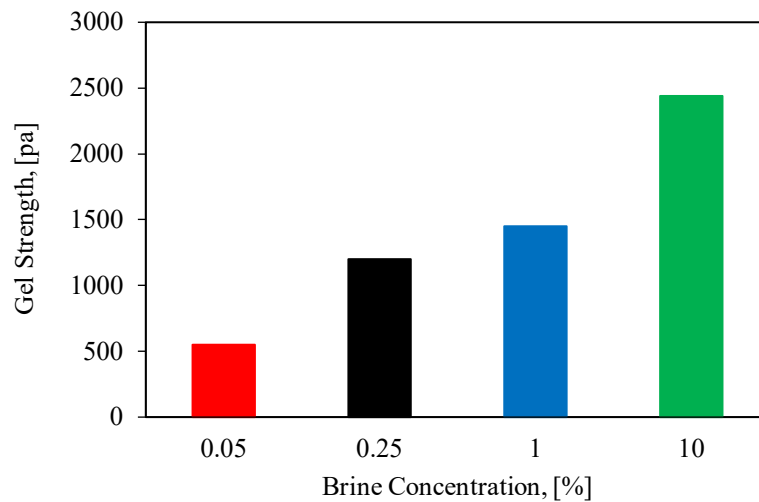


Figure 4. Gel strength at different brine concentration

## 4.2. PLUGGING EFFICIENCY RESULTS

In this section, the ability of the gel to plug cement channels and control fluids migration was tested using the gel-cement plugging efficiency setup (see Figure 2).

**4.2.1 Polymer Gel-Plugging Efficiency Results.** Water Breakthrough Pressure Measurements. The brine was first injected in four channels of 1.5 mm in diameter. The brine injection pressure was as low as 0.5 psi at flow rate of 2 ml/min as shown in Figure 5. After that the gel was injected in the channels until it reached a stable pressure of around

266 psi. After injecting around 49 pore volume of brine, the brine passed the gel at breakthrough pressure of 200 psi.

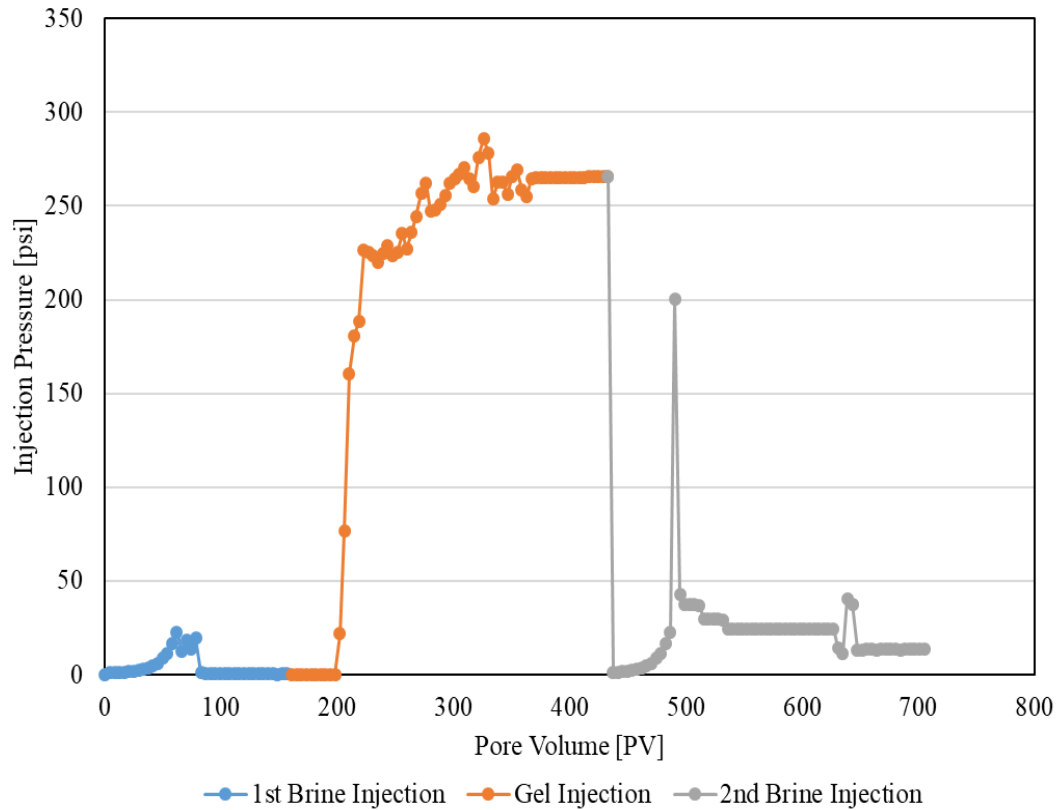


Figure 5. Injection pressure in 4 channels 1.5 mm

In the second test, the brine was first injected in a core with four channels of different sizes 1.5 and 1.15 mm in diameter. The brine injection pressure was as low as 6.1 psi at flow rate of 2 ml/min as shown in Figure 6. After that the gel was injected in the channels until it reached a stable pressure of 301 psi. After injecting around 90 pore volume of brine, the brine passed the gel at breakthrough pressure of 163.9 psi. These results show a significant effect of the size of the channels as the gel injection pressure increased by

291%. It is important to point out that the artificial channels created in this core sample were uniform.

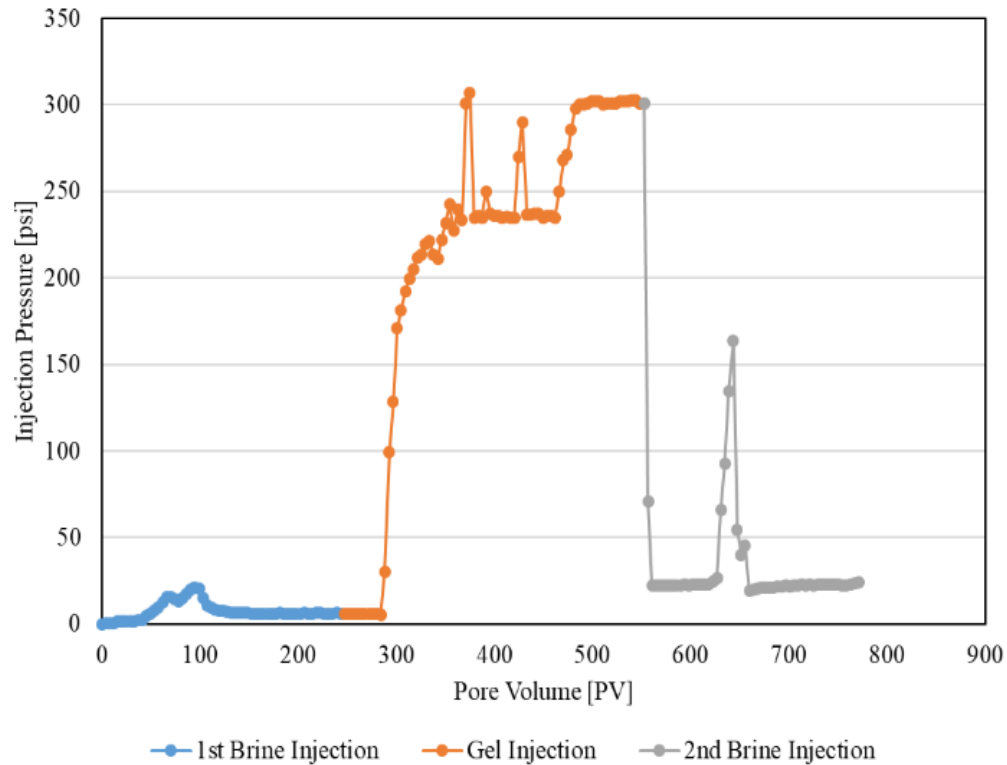


Figure 6. Injection pressure in 4 uniform channels 1.5 & 1.15 mm

In the third test, the brine was first injected in a core with four channels of different sizes (1.5 and 1.15 mm) distributed non-uniformly. The brine injection pressure was 22.9 psi at flow rate of 2 ml/min, which is higher than the injection pressures of the formal test. This can be seen in Figure 7. After that the gel was injected in the channels until it reached a stable pressure of 381 psi. After injecting around 128 pore volume of brine, the brine passed the gel at breakthrough pressure of 270 psi. These results show a significant effect of the size of the channels as the gel injection pressure increased by 266.4%.

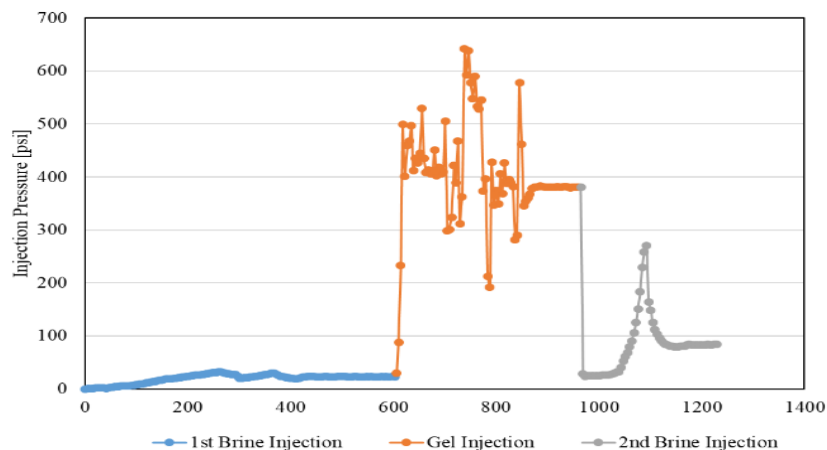


Figure 7. Injection pressure in 4 non-uniform channels 1.5 & 1.15 mm

Figure 8 provides information about the injectivity of the channels prior to gel injection for the three cases presented before gel treatment. The strength of the gel that was used is 1450 pa. The brine stable pressures before injecting the gel for all the channels has increased from 0.5 psi to 22.9 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the brine injection pressure.

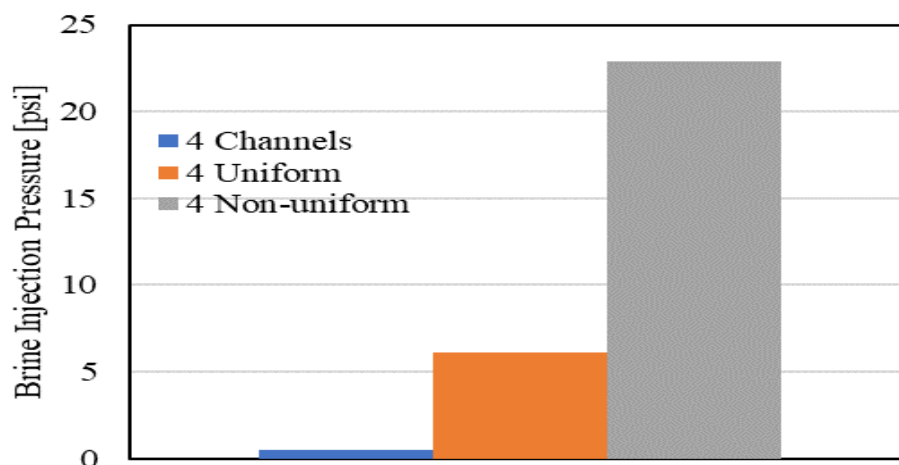


Figure 8. Stable brine injection pressures before gel treatment

Figure 9 provides information about the gel injection pressure for the three cases presented. The strength of the gel that was used is 1450 pa. The gel stable pressures after injecting the brine for all the channels has increased from 266 psi to 381 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the gel injection pressure.

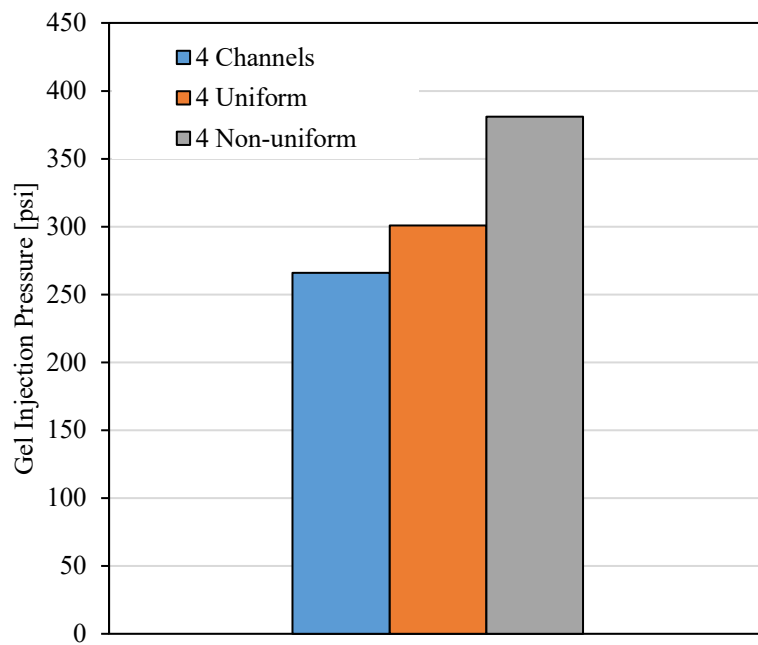


Figure 9. Stable particle gel injection pressures after brine injection

Figure 10 provides information about the breakthrough pressures for the three cases presented. The strength of the gel that was used is 1450 pa. The brine stable pressures after injecting the gel for all the channels has increased from 13.5 psi to 83.9 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the gel injection pressure.



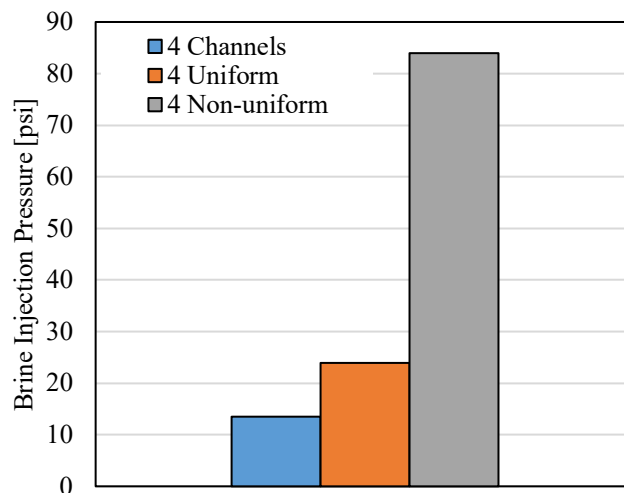


Figure 10. Stable brine injection pressures after gel treatment

Oil Breakthrough Pressure Measurements. The oil was first injected in four channels of 1.5 mm in diameter. The oil injection pressure was as low as 15.7 psi at flow rate of 2 ml/min as shown in Figure 11. After that the gel was injected in the channels until it reached a stable pressure of around 307 psi. After injecting around 37 pore volume of oil, the oil passed the gel at breakthrough pressure of 247 psi.

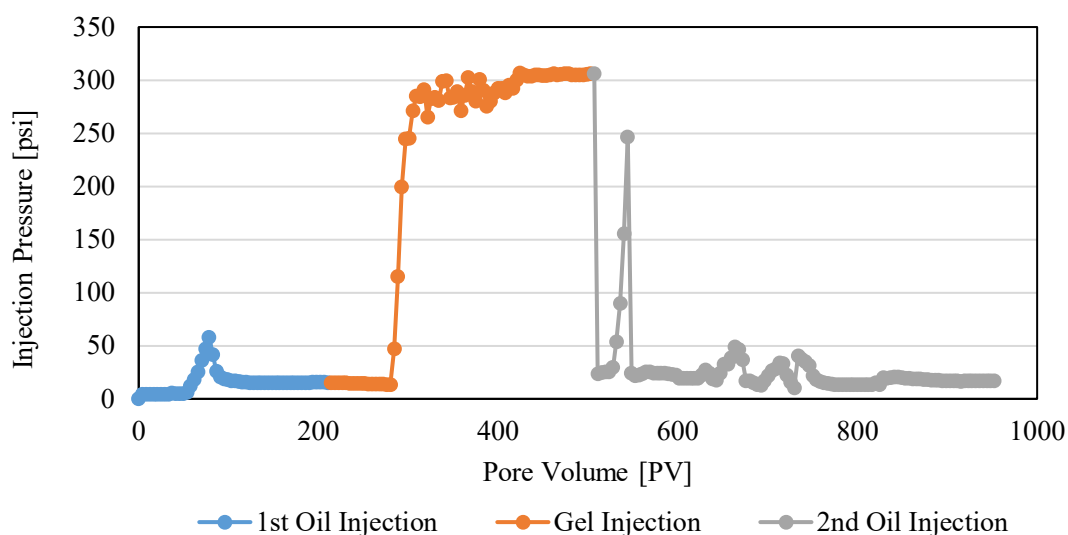


Figure 11. Oil injection in 4 channels 1.5 mm

In the second test, the oil was first injected in a core with four channels of different sizes 1.5 and 1.15 mm in diameter. The oil injection pressure was as low as 26.7 psi at flow rate of 2 ml/min as shown in Figure 12. After that the gel was injected in the channels until it reached a stable pressure of 537 psi. After injecting around 70 pore volume of oil, the oil passed the gel at breakthrough pressure of 359 psi. These results show a significant effect of the size of the channels as the gel injection pressure increased by 57%. It is important to point out that the artificial channels created in this core sample were uniform.

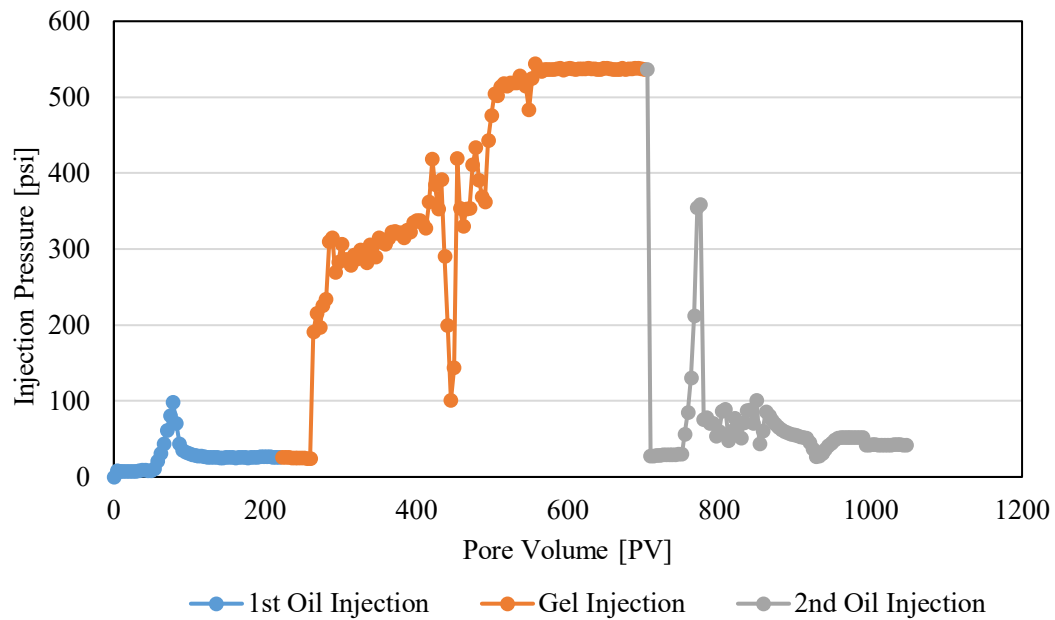


Figure 12. Oil injection in 4 uniform channels 1.5 & 1.15 mm

In the third test, the oil was first injected in a core with four channels of different sizes (1.5 and 1.15 mm) distributed non-uniformly. The oil injection pressure was 42 psi at flow rate of 2 ml/min, which is higher than the injection pressures of the formal test.

This can be seen in Figure 13. After that the gel was injected in the channels until it reached a stable pressure of 580 psi. After injecting around 161 pore volume of oil, the oil passed the gel at breakthrough pressure of 711 psi. These results show that the channels with non-uniform pattern has increased the gel injection pressure slightly.

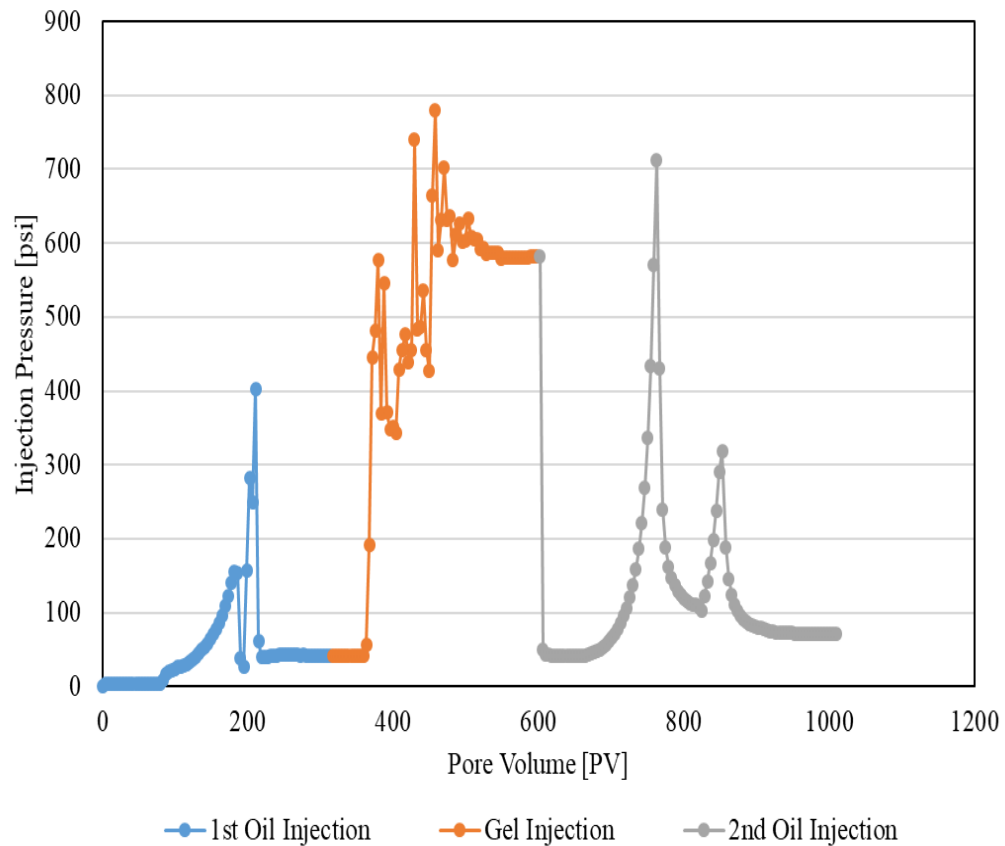


Figure 13. Oil injection in 4 non-uniform channels 1.5 & 1.15 mm

Figure 14 provides information about the injectivity of the channels prior to gel injection for the three cases presented before gel treatment. The strength of the gel that was used is 1450 pa. The oil stable pressures before injecting the gel for all the channels has

increased from 15.3 psi to 41.9 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the oil injection pressure.

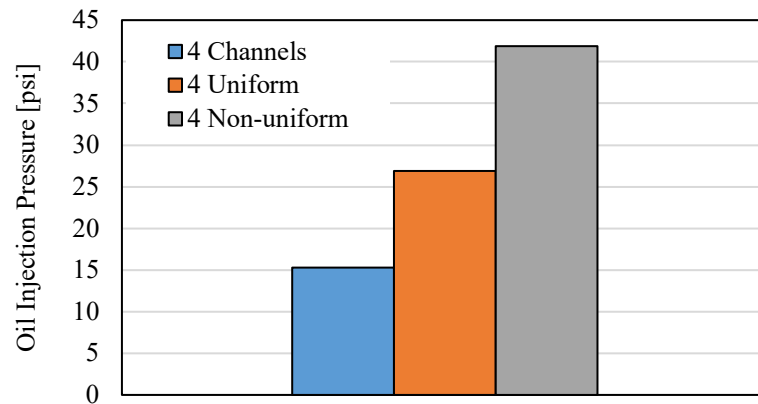


Figure 14. Stable oil injection pressures before particle gel treatment

Figure 15 provides information about the gel injection pressure for the three cases presented. The strength of the gel that was used is 1450 pa. The gel stable pressures after injecting the oil for all the channels has increased from 306 psi to 582 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the gel injection pressure.

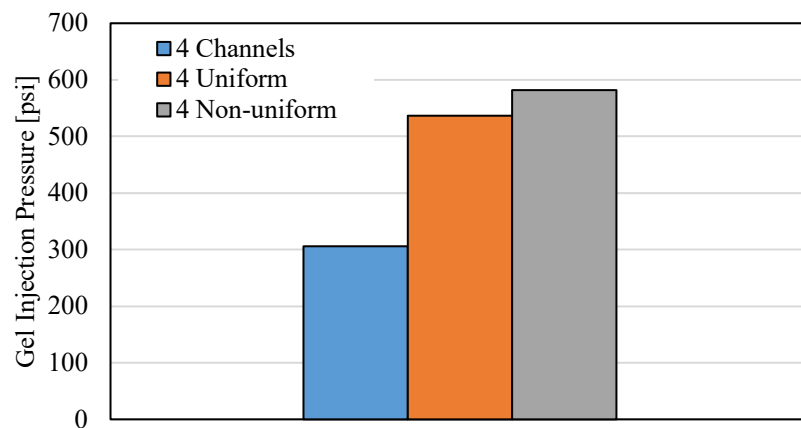


Figure 15. Stable particle gel injection pressures after oil injection

Figure 16 provides information about the breakthrough pressures for the three cases presented after gel treatment. The strength of the gel that was used is 1450 pa. The oil stable pressures after injecting the gel for all the channels has increased from 17 psi to 71.6 psi. These results indicate that the heterogeneity of the channels has the greatest effect on the gel injection pressure.

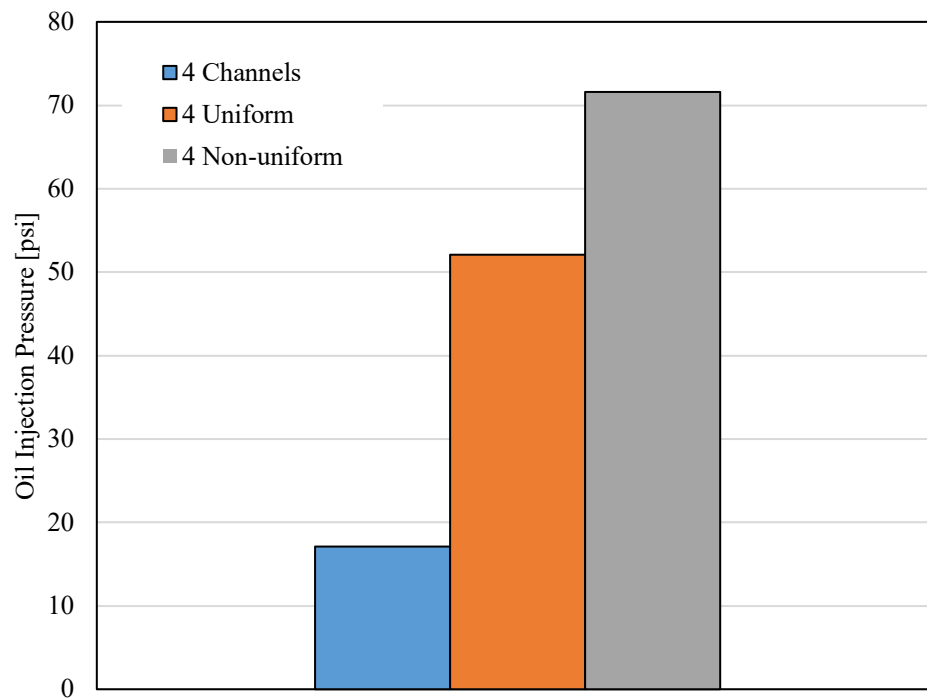


Figure 16. Stable oil injection pressures after particle gel treatment

Water Residual Resistance Factor. The particle gel blocking efficiency decreased when the heterogeneity of the channels increased. The results suggest that the particles gel can provide a 96.3% plugging in the 1.5 mm channels, as compared to 74.4% plugging for particles gel in the uniform 1.5 and 1.15 mm channels as listed in Table 2. These findings indicate that the plugging efficiency of the particle gel is affected significantly by the size of the channels.

Table 2. A summary of Frrw and blocking efficiency

<b>Channel Type</b>	1.5 mm	Uniform	Non-uniform
<b>Frrw [fraction]</b>	27	3.91	3.66
<b>Blocking Efficiency [%]</b>	96.29	74.42	72.67

Oil Residual Resistance Factor. The particle gel blocking efficiency increased when the heterogeneity of the channels increased. The results suggest that the particles gel can provide a 9.9% plugging in the 1.5 mm channels, as compared to 48.18% plugging for particles gel in the uniform 1.5 and 1.15 mm channels as listed in Table 3. These findings indicate that the plugging efficiency of the particle gel is very weak when used against oil. These results prove that the particles gel is a good candidate for conformance control.

Table 3. A summary of Frro and blocking efficiency

<b>Channels Type</b>	1.5 mm	Uniform	Non-uniform
<b>Frro [fraction]</b>	1.11	1.93	1.70
<b>Blocking Efficiency [%]</b>	9.9	48.18	41.17

## 5. DISCUSSION

Cement can fail in the annuli of the wellbore due to several reasons and the channels that are created can have different shapes and sizes. The performance of the gel as a sealant

can be affected by the injection fluids, the size, and geometry of the channels. Thus, it is essential to study the effects of the size and the geometry of the channels on the sealant before applying any remedial job. In this section, a comparison and analysis are presented to point out the effects of cement channels size and geometry on the gel plugging efficiency for both oil and brine. Gel with strength of 1450 pa was used for this investigation.

The results of particle gel injection after water was the highest when the gel was injected in the non-uniform channeled core. The injection pressure decreased dramatically from uniform channels with different sizes to uniform channels with the same sizes as shown in Figure 17. Also, the results of particle gel injection after oil was the highest when the gel was injected in the non-uniform channeled core. The injection pressure decreased dramatically from uniform channels with different sizes to uniform channels with the same sizes as shown in Figure 18. In addition, the injection pressures of particles gel after brine were lower than that of the injection pressures of particles gel after oil.

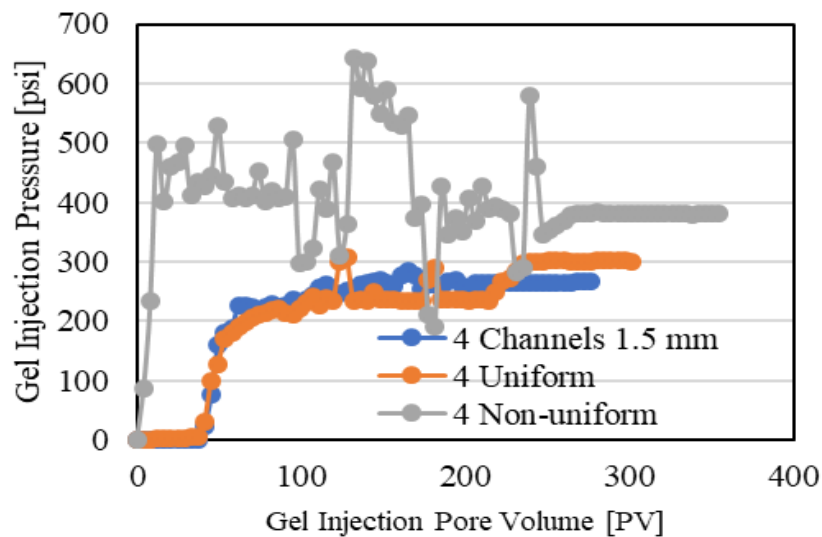


Figure 17. Particle gel injection pressure after brine injection

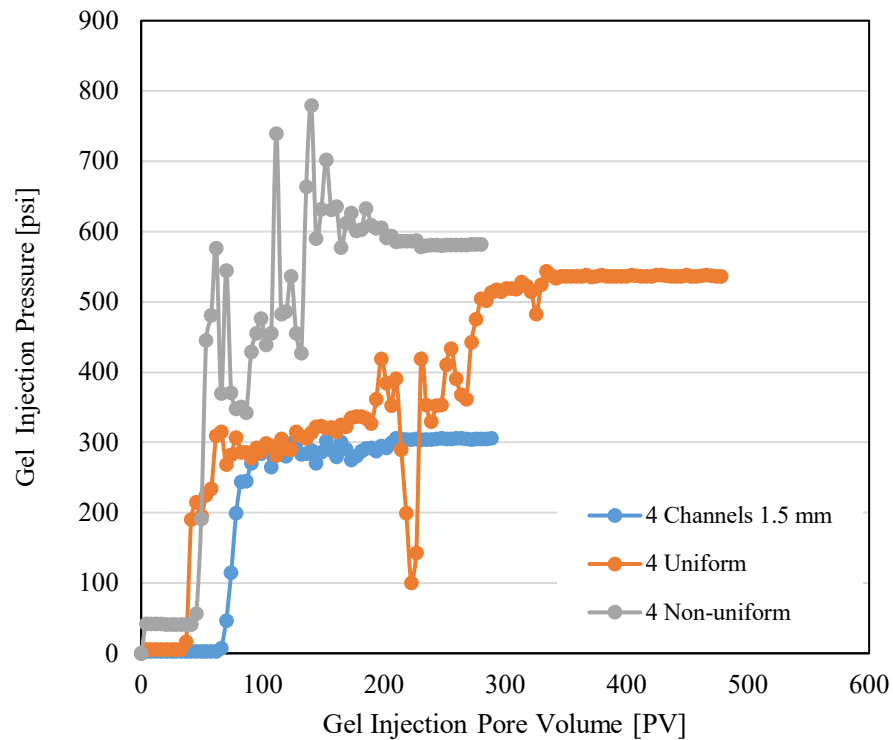


Figure 18. Particle gel injection pressure after oil injection

Water Breakthrough Pressure Measurements. After gel placement within the cement channels, brine was injected with constant flowrate to characterize the particle gel blocking behavior against brine. Figure 19 shows the breakthrough pressures of the brine with different channels cores.

Oil Breakthrough Pressure Measurements. After gel placement within the cement's channels, oil was injected with constant flowrate to characterize the particle gel blocking behavior against oil. Figure 20 shows that the gel in the non-uniform channels resulted in the highest pressure to break (712 psi). This peak indicates the point at which gel failed and washout began to occur (Seright, 2003).



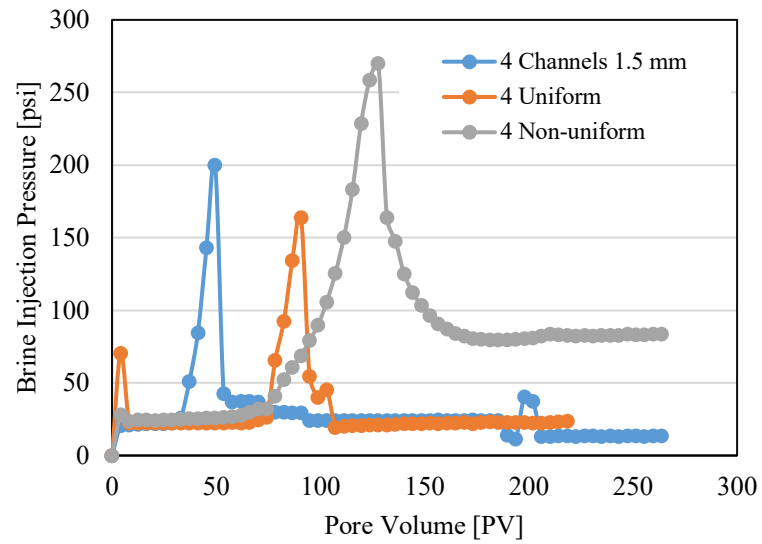


Figure 19. Brine injection pressure after particle gel treatment

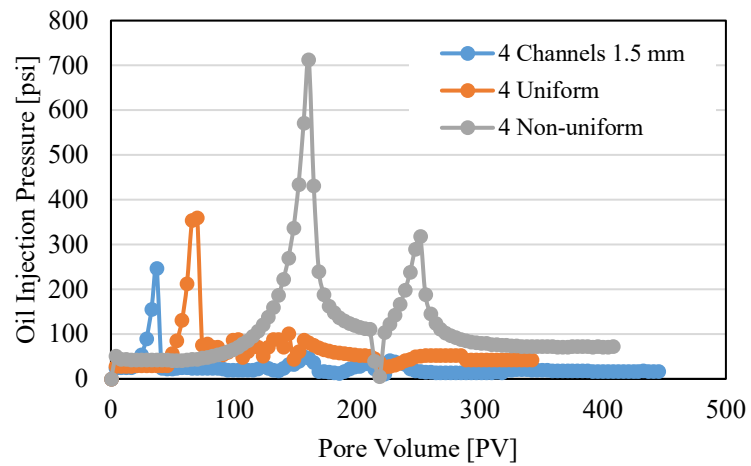


Figure 20. Oil injection pressure after particle gel treatment

## 6. CONCLUSIONS

By studying the effects of injecting particle gel in cement channels, several findings were obtained in this study. These findings are based on the analysis of gel-plugging efficiency experiments. The main conclusions are summarized below:

- The change in cement channel size has a very clear effect on the injection pressure of the gel particles.
- Increasing the heterogeneity of the cement channels will affect the fluid flow.
- Decreasing the cement channel size will increase the pressure of the gel injection.
- The gel plugging efficiency to water flow through the cement's fracture is controllable.
- Selecting the gel sealant material depends on the plugging efficiency.
- The breakthrough time and pressure were changed when the size of cement channels was changed.

### **ACKNOWLEDGEMENT**

The author wishes to thank King Abdulaziz University (KAU) for granting him his scholarship.

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### **III. THE APPLICATION OF EPOXY RESIN SEALANT TO REMEDIATE WATER AND OIL LEAKAGE IN CEMENT FRACTURES AND CHANNELS**

#### **ABSTRACT**

One of the most important operation in the oil field is the seal integrity of oil and gas wells. In the event that the integrity of the well is compromised during any period of the life of the well, the outcomes could involve serious ramifications for both the personnel and the equipment. Sealant material can be utilized to seal pathways that were created in the cement sheath. This study investigates the use of Novolac epoxy resin sealant for wellbore integrity applications. This work considers the impact of temperature on the curing time of the sealant, the ability of the sealant to plug the pathways, and the ability of the sealant to stop leakages. The lab experiments incorporate rheological examination, curing time estimations, blocking efficiency, and mechanical properties. The findings of this investigation demonstrate that this sealant has Newtonian rheological behavior, the ability to penetrate small gaps, the capacity to oppose differential pressure as high as 1800 psi, and compressive strength higher than 8931.3 psi. This work shows that Novolac epoxy resin sealant can be utilized successfully in plugging cement cracks and channels.

#### **1. INTRODUCTION**

After drilling the oil and gas wells and running the casing, cement is placed in the annulus of the casing and the drilled formation. This is called the primary cement operation. This cement may fail during any stage of the life of the well (Watson & Bachu, 2008). The

failure of the cement may result in fluid migration, which may compromise the integrity of the well. Wellbore integrity is defined as the “application of technical, operational and organizational solutions to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well” (NORSOK D-010, 2013). Fluids can migrate through pathways in the cement itself, between the cement and the formation, or between the cement and the casing. When the wellbore integrity is compromised, these pathways occur and formation fluids are allowed to migrate between formations and/or from the formation to the surface. Leakage of fluids may occur through the cement pathways during the drilling or production stage of the well or after the abandonment of the well, which may endanger the personnel and the environment (Davies et al., 2014; Alkhamis & Imqam, 2018; Ahdaya & Imqam 2019). The leakage of fluids along the interface between the wellbore and the formation is a primary concern in hydrocarbon recovery (Dusseault et al., 2000). Leakage pathways in the cement annulus can happen due to mechanical failures as a result of pressure and temperature cycles, and chemical degradation can occur due to the corrosive formation fluids (Liu et al., 2017) or due to improper slurry design. Improper slurry design includes the use of slurries with low densities, which may allow formation fluids to create channels in the cement; the use of slurries with uncontrolled fluid loss, which may affect the strength of the hardened cement; and the use of very brittle cement, which can break down as a result of casing contractions and expansions. In addition, failures can occur due to improper mud removal. The remaining mud on the surface of the formation and casing can prevent the cement from bonding with its surroundings, creating micro-annuli. These and similar failures create pathways for formation fluids through the cement barrier.

Cement squeeze remedial operation is commonly performed to seal cement leakages. However, the cement squeeze cannot be used effectively to mitigate and prevent leakage because of low cement injectivity, pressure restriction, pin-hole leakage, micro-channels and fractures inside the leaking formation, and micro-cracks within primary cement (Jones et al., 2014). To overcome these limitations, self-healing cement was developed (Cavanagh et al., 2007; Roth et al., 2008; Reddy et al., 2010). Self-healing cement enables an automatic repair when micro-annuli, an internal cement crack, or another flow path is created. However, these kinds of sealing materials still have some problems associated with their swelling chemical additives and other concerns related to their mechanical and thermal stability.

Today, epoxy resin materials are being used to plug the cement pathways. This type of sealant seems to work effectively, but it needs more investigation to ensure proper placement and performance. Epoxy resin was first offered commercially in 1946 (Dewprashad & Eisenbraun, 1994). Epoxy is a product of the reaction between epichlorohydrin and bis-phenol A, and a common hardener is diethylenetriamine (Kabir, 2001). The viscosity of the epoxy resin can be altered using different types of diluents, such as ethylene glycol mono-butyl ether (Hakiki et al., 2015). The epoxy resin as a material offers many advantages over cement such as good viscosity. In addition, it is a solids-free material that can penetrate small gaps (Alsaihati et al., 2017; Todd et al., 2018), has good wetting and adhesive properties for mineral surfaces (Brooks et al., 1974), especially silica surfaces (Shaughnessy et al., 1978), has good density and tunable setting time (Sanabria et al., 2016), and exceptional resistance to contamination (Perez et al., 2017). These properties were the main motivation for this study.



In this study, the viscosity of Novolac epoxy resin is measured using a high tech rheometer, the effect of the temperature on the curing of the epoxy resin was investigated using the rheometer, the blocking performance was measured using a special setup, the compressive strength was measured, and the bonding property of the sealant to steel was studied.

## 2. EXPERIMENTAL DESCRIPTION

### 2.1. MATERIALS

Class-H Cement. All the cement specimens that were used in this study were prepared using American Petroleum Institute Class H cement and distilled water. The specific gravity of the cement was found to be 3.18 using a gas pycnometer. The chemical composition of Class-H cement was obtained using X-ray fluorescence spectroscopy (XRF) and is listed in Table 1.

Table 1. The chemical composition of class-H cement

<b>Composition</b>	<b>CaO</b>	<b>SiO<sub>2</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>SO<sub>3</sub></b>
<b>Wt %</b>	65.72	20.36	6.19	3.17	2.26
<b>Composition</b>	<b>MgO</b>	<b>K<sub>2</sub>O</b>	<b>SrO</b>	<b>TiO<sub>2</sub></b>	<b>Other</b>
<b>Wt %</b>	1.32	0.43	0.21	0.16	0.18

Channeled Cement Core Preparation. In preparation, cement cores with channels, were filled with cement. Wires of 1.15 and 1.5 mm in diameter were placed in 1 in, diameter molds to create artificial channels in the cement cores. The cement was poured

into the molds and left for more than 72 hours to set. Some of the wires were placed uniformly, and some wires were placed non-uniformly to observe the difference between the two cases. Figure 2 illustrates the channeled cement core.

**Resin.** The resin used in this study consists of phenol, polymer with formaldehyde, and glycidyl ether, also known as Novolac epoxy resin. This type of resins is designed for good thermal stability and chemical resistance.

**Curing Agent.** The curing agent of the epoxy resin was 1,2 cyclohexanediamine B, which is an aliphatic hardener.

**The Sealant Chemical Reaction.** The sealant material used in this study consists of two components: the first one is the epoxy resin and the second one is the epoxy hardener. The resin is Novolac resin, which is a mixture of phenol, polymer with formaldehyde, and glycidyl ether resin. The hardener is 1,2 cyclohexanediamine B. The Novolac epoxy resins are specially considered to afford high chemical resistance and very good thermal stability. Figure 1 shows the chemical structure and the chemical reaction of each component.

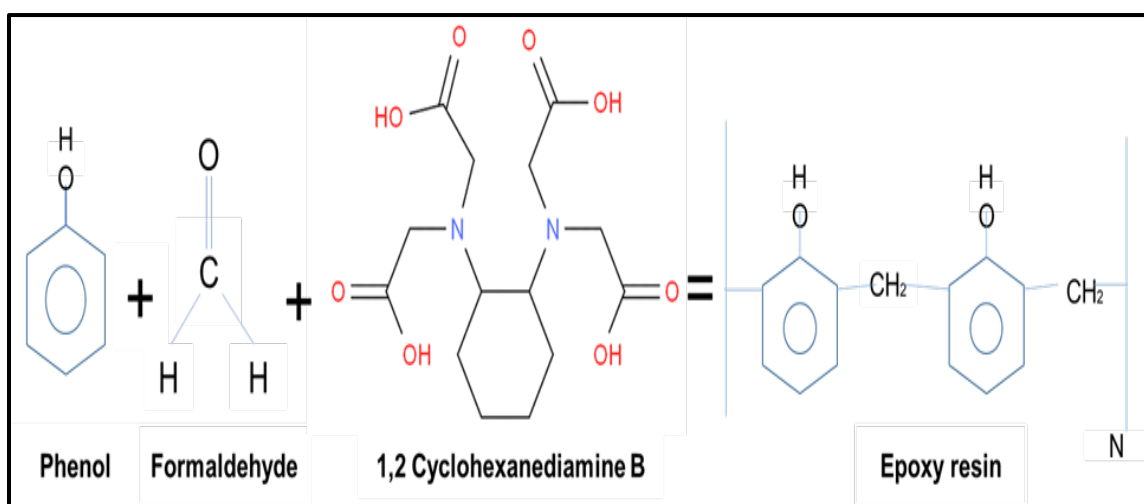


Figure 1. The sealant chemical reaction

Cement Paste and Cores Preparation. The cement slurry was mixed at room temperature using a two speed bottom-drive blender. A specific amount of water was poured in the blender, and then dry cement was added at a uniform rate while mixing at low speed for around 15 seconds. Then the blender was covered, and the mixing continued for extra 35 seconds at high speed (API RP 10B-2 2013). The cement slurry had a water/cement ratio (WCR) of 0.38 in accordance with the API specification 10A (API 2010).

Fractured Cement Cores Preparation. To prepare the fractured cement cores, one inch in diameter molds were cut into two halves (Figure 2). The cement was poured into each half and left for more than 72 hours to set. Then, two metal sheets with specific thickness (0.5 mm and 2.0 mm) were used to separate the two cement specimens to the targeted fracture width.

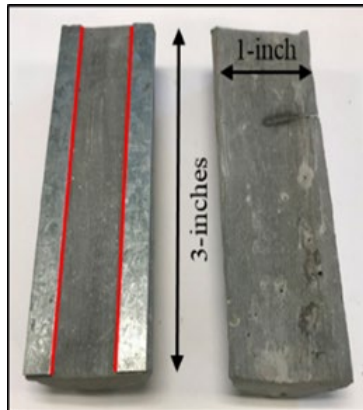


Figure 2. An illustration of fracture cement core

Channeled Cement Core Preparation. In preparation, cement cores with channels, were filled with cement. Wires of 1.15 and 1.5 mm in diameter were placed in 1 in,

diameter molds to create artificial channels in the cement cores. The cement was poured into the molds and left for more than 72 hours to set. Some of the wires were placed uniformly, and some wires were placed non-uniformly to observe the difference between the two cases. Figure 3 illustrates the channeled cement core.



Figure 3. An illustration of channeled cement core

**Epoxy Resin Preparation.** To formulate the epoxy resin system, a ratio 1 to 2 of resin to hardener was used. At room temperature, the materials were mixed at a low shear rate using a magnetic stirrer until the mixture became homogenous.

### **3. EXPERIMENTAL METHODOLOGY**

In this section of the paper, the description of all the experiments conducted in this work is presented. The experiments include rheological behavior, curing measurements, blocking performance, bonding strength, and compressive strength.

### **3.1. RHEOLOGICAL MEASUREMENTS**

These measurements are important for this study to understand the behavior of the sealant material. In these measurements, the epoxy resin samples were mixed at room temperature. An Anton Paar Rheometer (DSR) with a disposal parallel plates system was used to measure the rheological behavior of the sealant material. Samples of 1.0 ml of the sealant were placed on the lower plate of the rheometer and the upper plate was then lowered to a gap of around 1.0 mm.

### **3.2. ISOTHERMAL CURING MEASUREMENTS**

These measurements are executed to estimate the curing time of the sealant material to define its workability. For these measurements, an oscillatory test using the rheometer was performed at angular frequency of 10 rad/s and the complex viscosity increase with time was recorded at different temperatures. Disposal parallel plates were used as the test was run until the material reached its solid state.

### **3.3. BLOCKING PERFORMANCE MEASUREMENTS**

The blocking performance measurements were conducted to study the ability of the sealant to block the cement cracks and channels. The sealant was injected in the cement gaps and left for 24 hours to cure at room temperature. After that, the cement cores were placed in a core holder, and then water and oil were injected at differential pressures up to 1800 psi. Figure 4 is an illustration of the setup that has been used for this measurement.

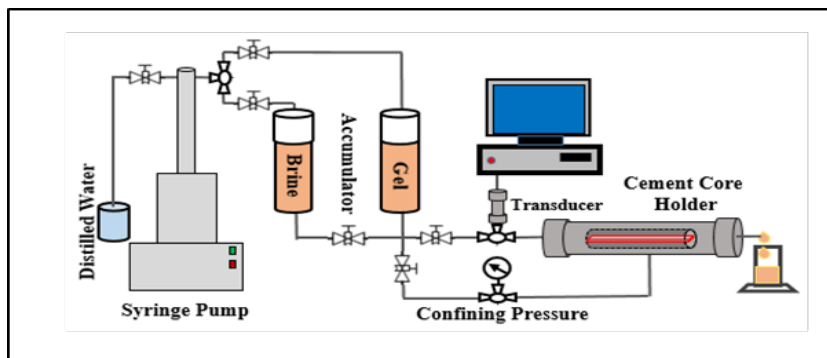


Figure 4. Blocking performance setup

### 3.4. COMPRESSIVE STRENGTH MEASUREMENTS

In this test, the compressive strength measurements were executed to determine the strength of the Novolac epoxy resin. The compressive strength of the sealant is an indication of the ability of the sealant to resist wellbore conditions. For this test, the epoxy resin was mixed at room temperature and poured into 2" × 4" cylindrical molds. A caliper was used to measure the height and diameter of the sample, and the surface area was calculated. A hydraulic press was used to measure the force needed to break down the samples, as shown in Figure 5.



Figure 5. The compressive strength equipment

### 3.5. BONDING STRENGTH MEASUREMENTS

The bonding strength of the Novolac epoxy resin with steel was measured by the hydraulic press, as shown in Figure 4. In this test, the epoxy resin was mixed at room temperature and poured in a cylindrical mold that contains steel. The shape of the steel is shown in Figure 6. The test was performed after 24 hours of curing. The compression load pushed the sample until the debonding occurred. The load and displacement between the two materials was measured.

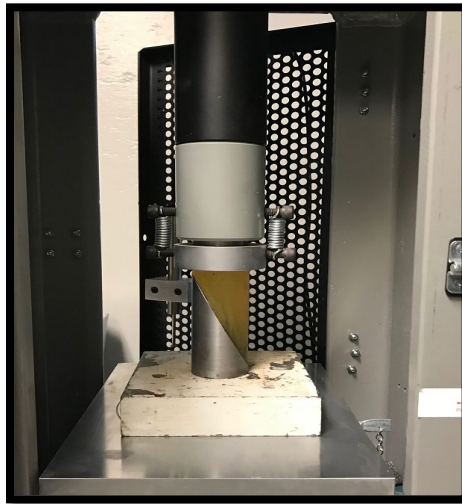


Figure 6. The bonding strength equipment

## 4. RESULTS AND ANALYSIS

In this section of the paper, the results of each experiment are presented and analyzed according to their importance in the application of the sealant.

#### 4.1. RHEOLOGICAL MEASUREMENTS RESULTS

First, the viscosity of the resin was measured using the rheometer. The viscosity of the sealant resin was found to be in the range of 1699 cp at a high shear rate to 1657 cp at a low shear rate. The behavior of the sealant material under the accelerated shear rate was shear-thinning, meaning that increasing the shear rate decreased the viscosity of the resin. The measurements were conducted at room temperature and atmospheric pressure. For remedial application, this is an effective sealant material as it supports the main goals of sealing cement fractures and channels. The structure change was observed by taking the viscosity measurements of two ramps up and down. Figure 7 shows these results. In this study, the behavior of the sealant material was Newtonian-like.

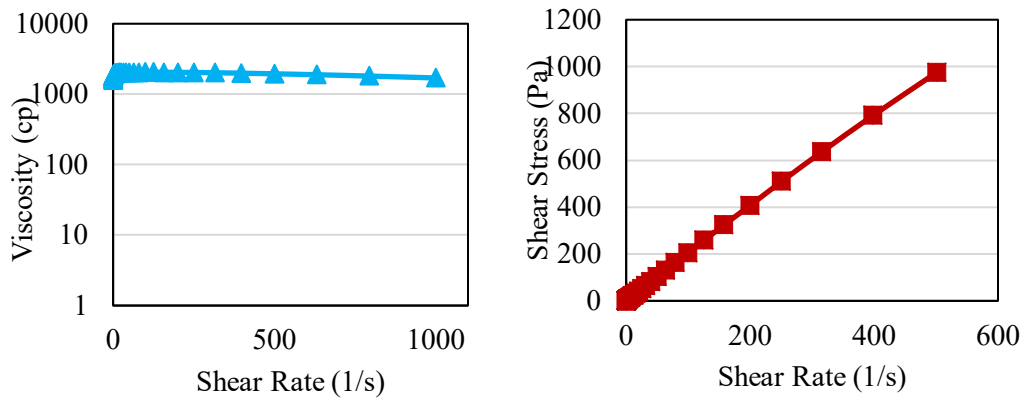


Figure 7. The viscosity results of the sealant

#### 4.2. CURING TIME MEASUREMENTS RESULTS

In this test, we used the parallel plates to add the sealant material and start to take the measurements. Curing measurements were conducted at three constant temperatures: 24°C, 60°C and 100°C. The objectives of these measurements are to determine the



workability time of the sealant at different temperatures and to study the effect of temperature on the curing process of the sealant. At 24°C, the sealant complex viscosity increased steadily for around 4 hours. This time could be the gelling time. The material started to transfer to solid after around 3 hours and 45 minutes. When the system cured for around 6 hours, the complex viscosity was around 10,000,000 cp, as shown in Figure 8. At 60°C, the same behavior was seen except for the curing, which became shorter as the sealant was fully cured after one hour. These measurements are crucial to optimize the placement of the sealant in the cement fractures and channels. One of the important things that we need to know is the curing time. Increasing or decreasing the curing time will affect the injection of the sealant material and cause it to take either a longer or shorter time. Plugging the coil tubing or not reaching the target zone will decrease the sealant material or plug the equipment. To be successful, a remedial job needs to optimize the curing time. The exact gelling time of the epoxy sealant material is important; when the deferent temperature is increased, the time will decrease prior to the remedial job at the surface.

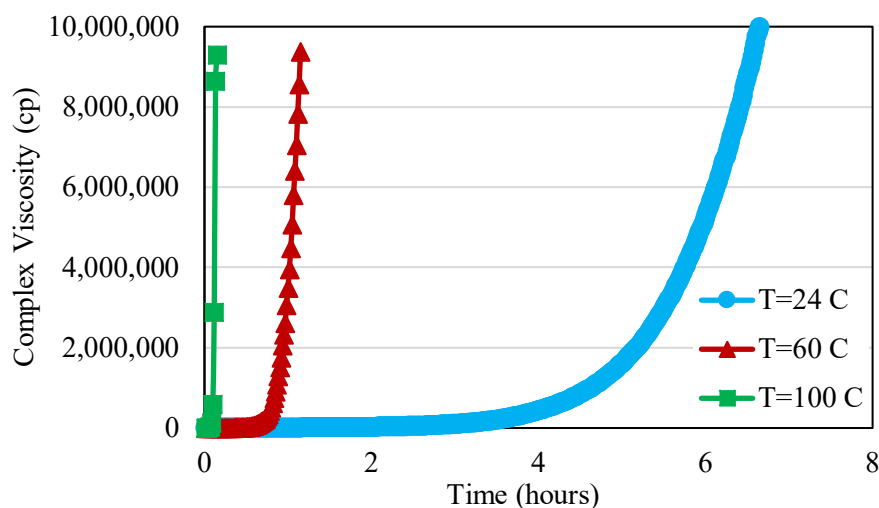


Figure 8. The curing results of the sealant at different temperatures

#### 4.3. BLOCKING PERFORMANCE MEASUREMENTS RESULTS TO WATER

Before the placement of the sealant in cracks and channels of the cement cores, the brine was produced at low pressure at constant flow rate as shown in Figure 9 and Figure 11. After the placement of the sealant in the cracks and channels of the cement cores, the cores were left to cure for 24 hours. Next, the core was tested against the water flow. The injection pressure of the water started by 295 psi in the cement channel core sample. No water was produced when the pressure increased to 1000 psi for more than 14 minutes. Then, the pressure was increased to 1800 psi and left for around 8 minutes. Again, no water was observed at the outlet of the core, as shown in Figure 10. Figure 12 shows the injection pressure of the second sample, which was cement cores with cracks. The pressure increased from 500 psi to 1800 psi, and again, no water was produced. These results indicate full plugging of the channels and the cracks in the cement cores.

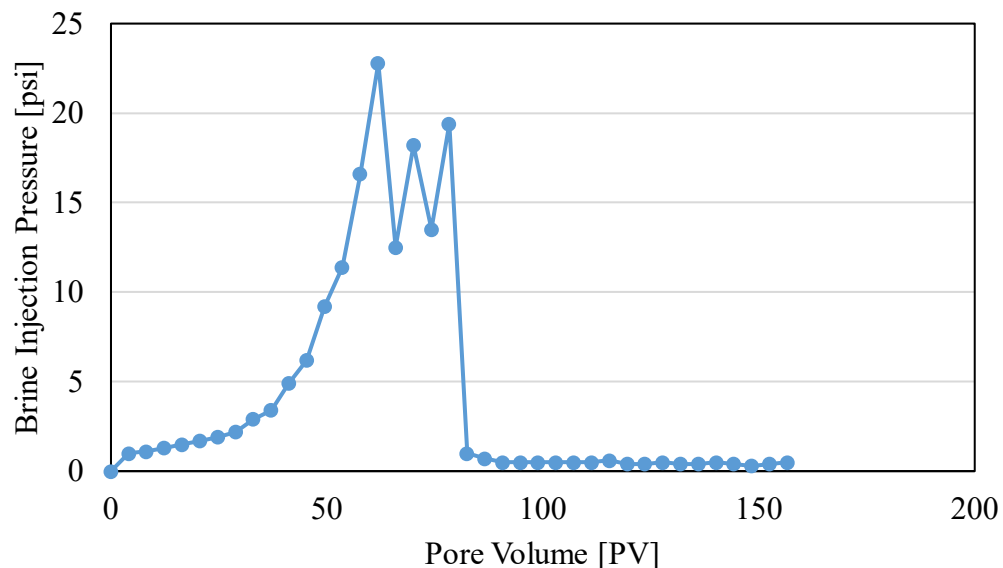


Figure 9. Water injection before placement of sealant in channels

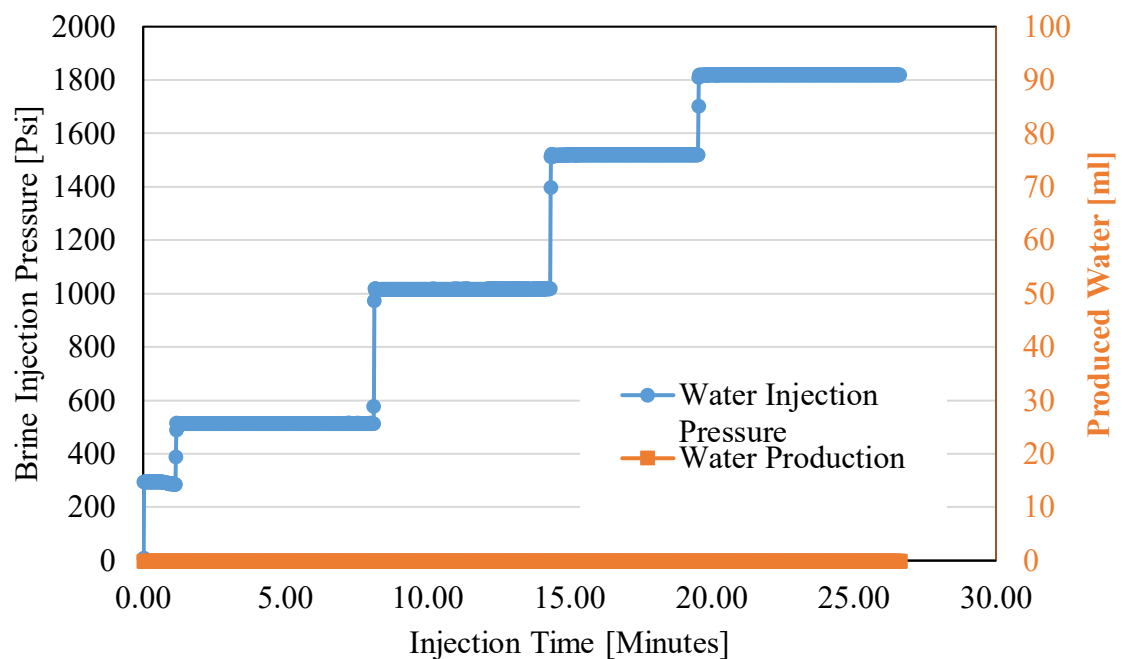


Figure 10. Water injection after placement of sealant in channels

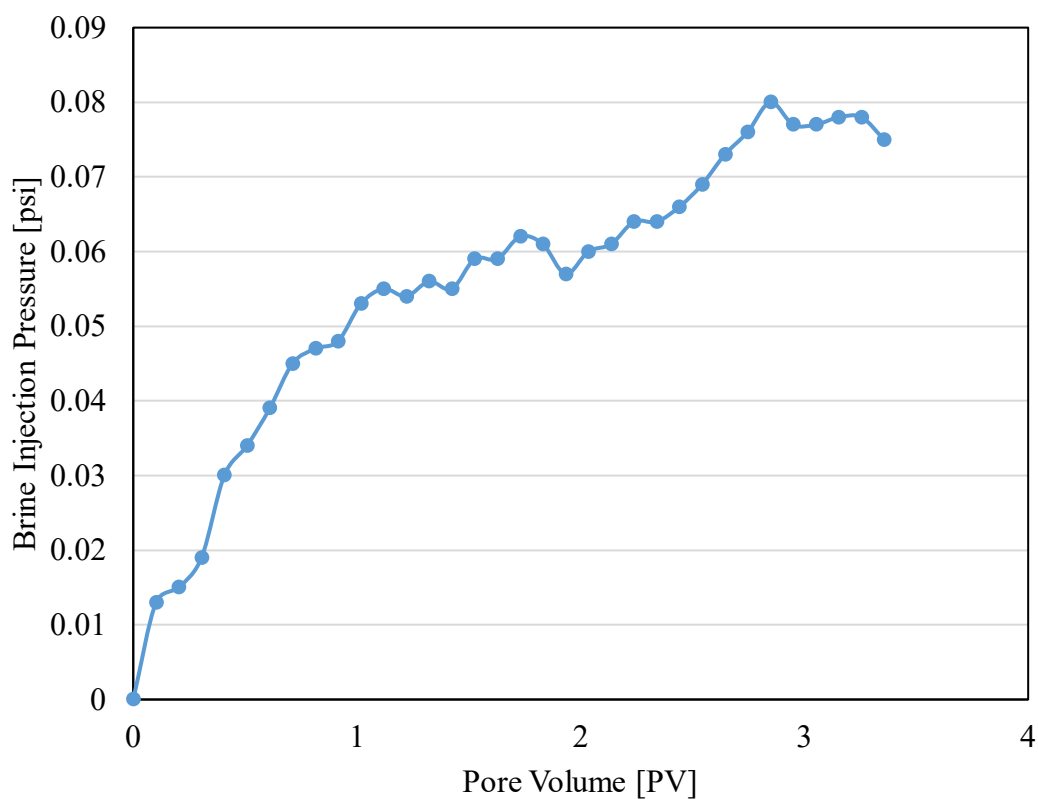


Figure 11. Water injection before placement of sealant in fracture

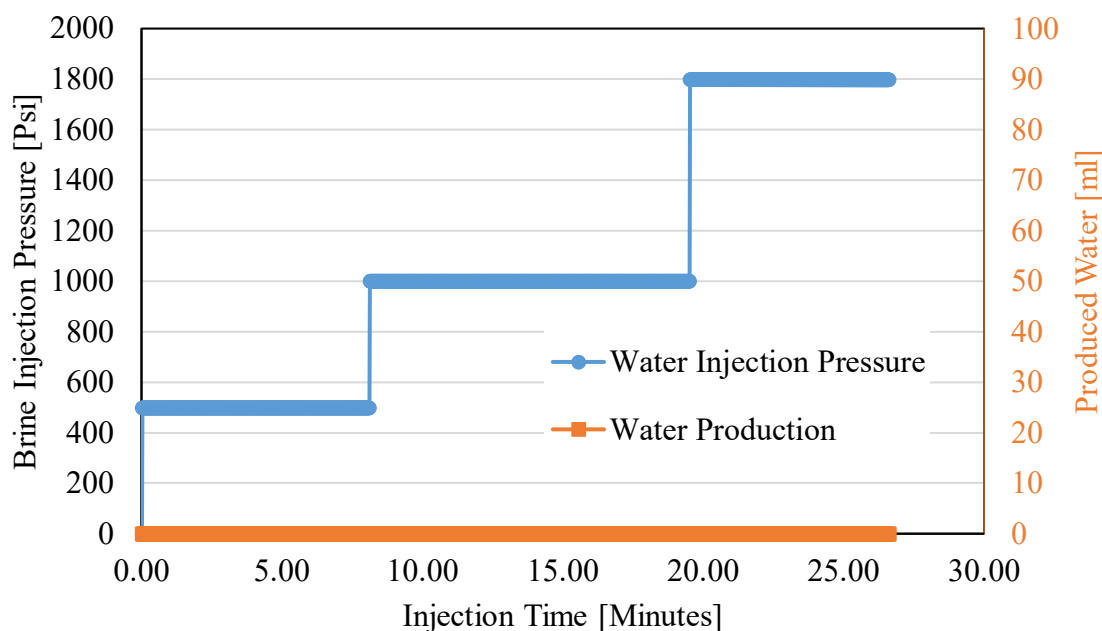


Figure 12. Water injection after placement of sealant in fracture

#### 4.4. BLOCKING PERFORMANCE MEASUREMENTS RESULTS TO OIL

Before the placement of the sealant in cracks and channels of the cement cores, the oil was produced at low pressure at constant flow rate as shown in Figure 13 and Figure 15. After the placement of the sealant in the cracks and channels of the cement cores, the cores were left to cure for 24 hours. Then, the core was tested against oil flow. By starting to injection oil pressure from 500 psi and increased to 2000 psi in the first sample which is the cement channels core sample for more than 25 minutes. No oil was produced as shown in Figure 14. When changing the core sample to cement cracks core sample and starting to injected the oil from 400 psi until 2000 psi the result was no oil produced as shown in Figure 16. This experiment proves the ability of this sealant to completely seal cement channels and cracks in a differential pressure up to 2000 psi and to reduce the amount of produced water or oil under higher pressures.

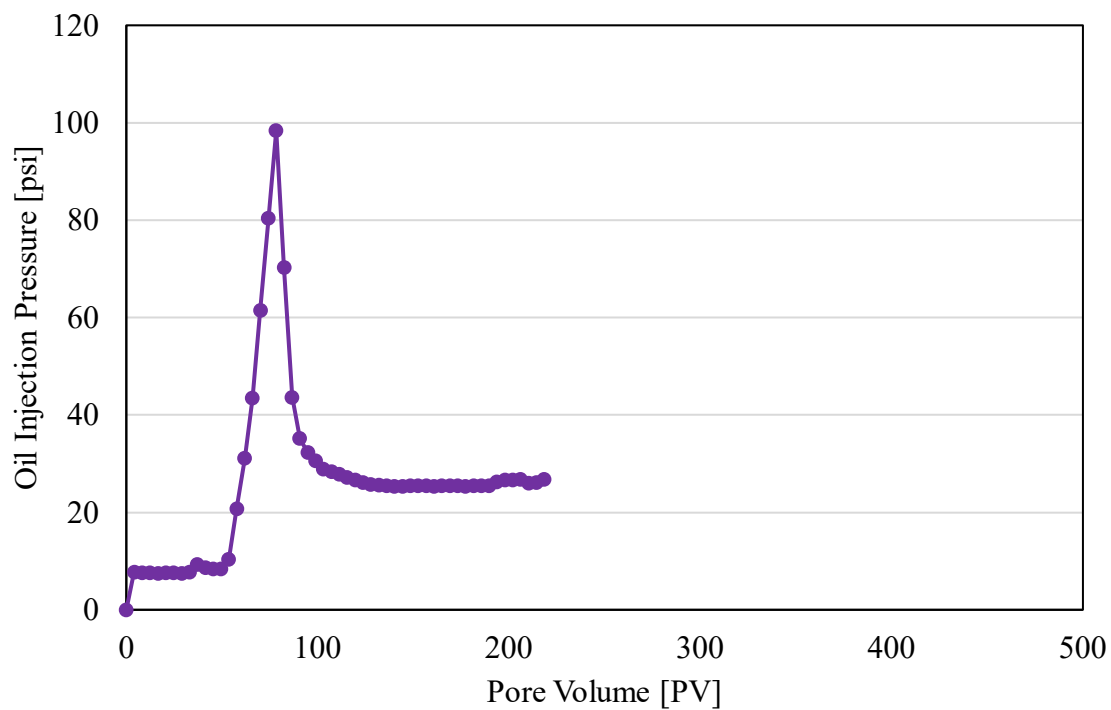


Figure 13. Oil injection before placement of sealant in channels

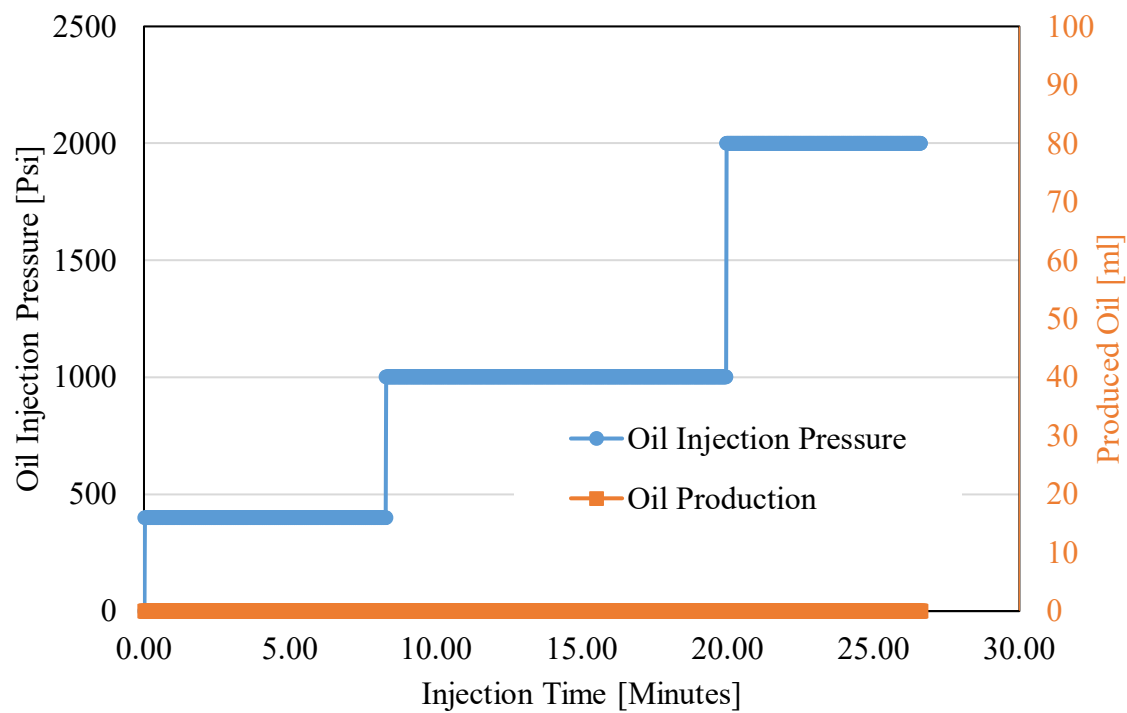


Figure 14. Oil injection after placement of sealant in channels

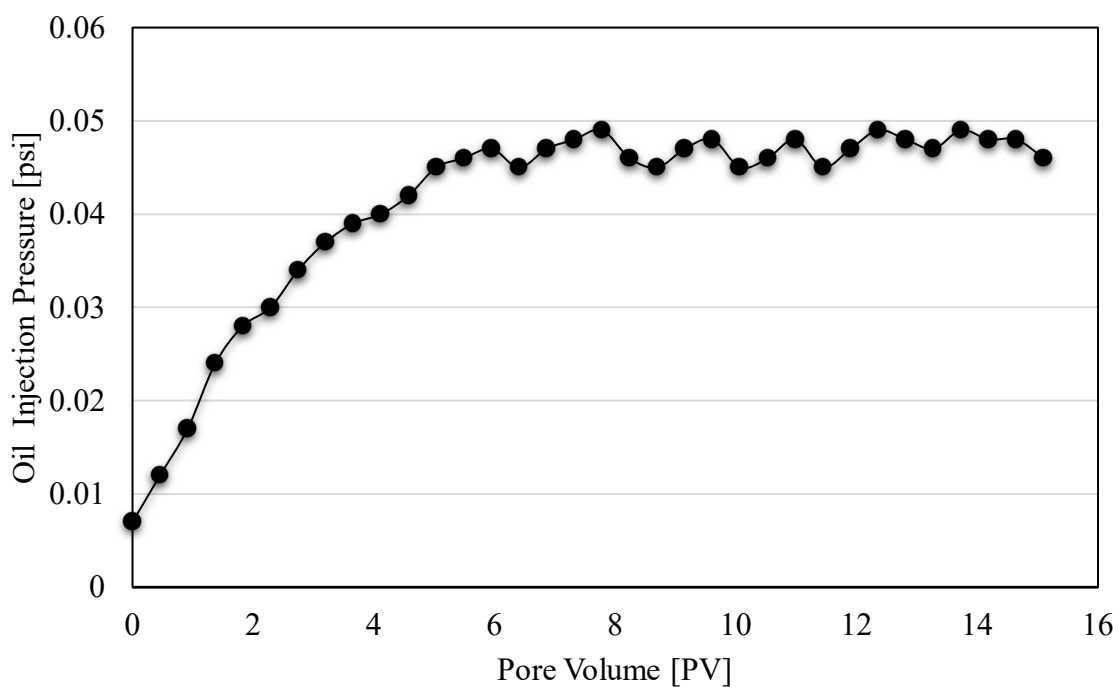


Figure 15. Oil injection before placement of sealant in fracture

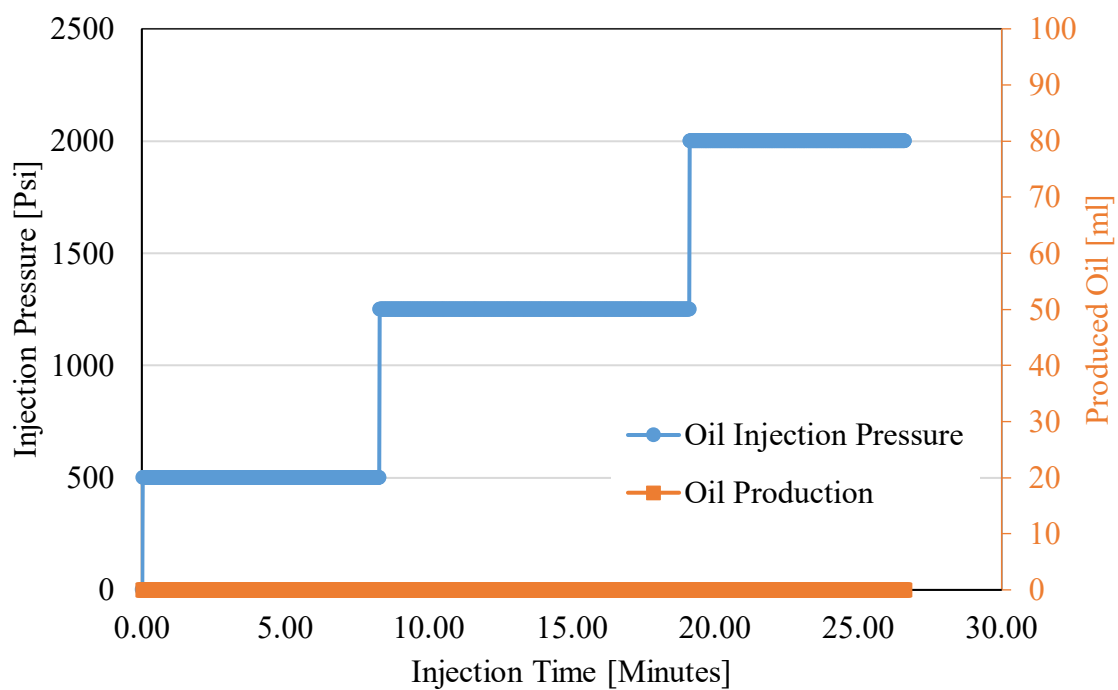


Figure 16. Oil injection after placement of sealant in fracture

## 4.5. MECHANICAL MEASUREMENTS RESULTS

**4.5.1. Compressive Strength Results.** For the compressive strength of the sealant, a load was applied to the cubic mold until the compressive strength reached values greater than 8931.3 psi without failure, as shown in Table 3. The cubic mold did not fail under this high load and returned to its original shape, showing high ductility and strength. Figure 17 shows the load rate of the Novolac epoxy sealant material.

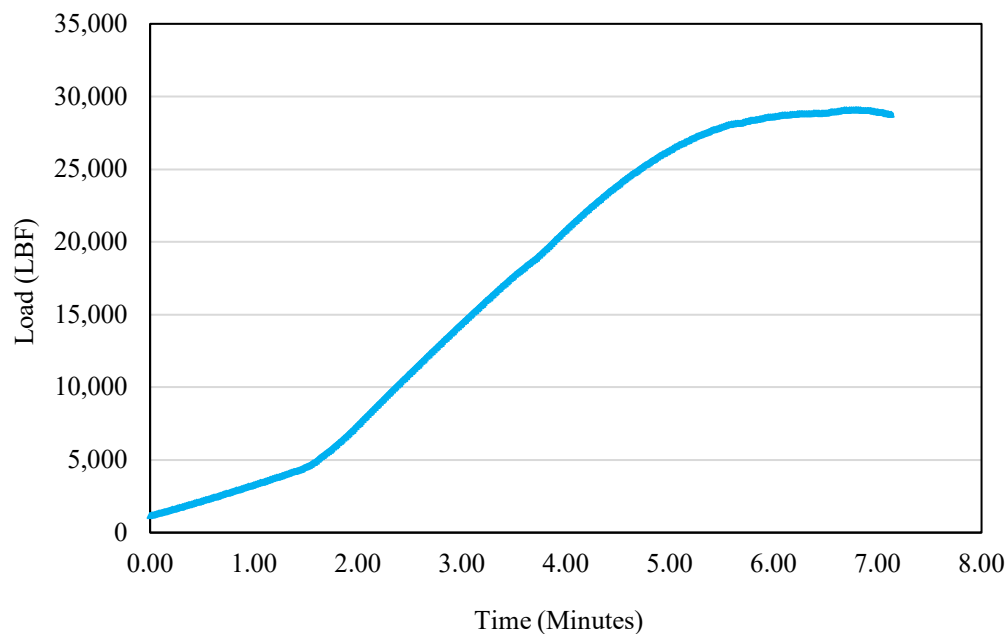


Figure 17. Load rate of compressive strength measurement

Table 2. Compressive strength

Compressive Strength (psi)	Load (lbf)	Diameter (in)	Length (in)	Cross Sectional Area (in <sup>2</sup> )
8931.3	29116	2.04	3.7	3.26

**4.5.2. Bonding Strength Results.** To test the bonding strength of the sealant with steel, a load was applied to the cylindrical mold until the Novolac epoxy resin separates from the steel, and it was 1587 lbf (as shown on Table 4) with all dimensions. Figure 18 shows the load at which the debonding occurred.

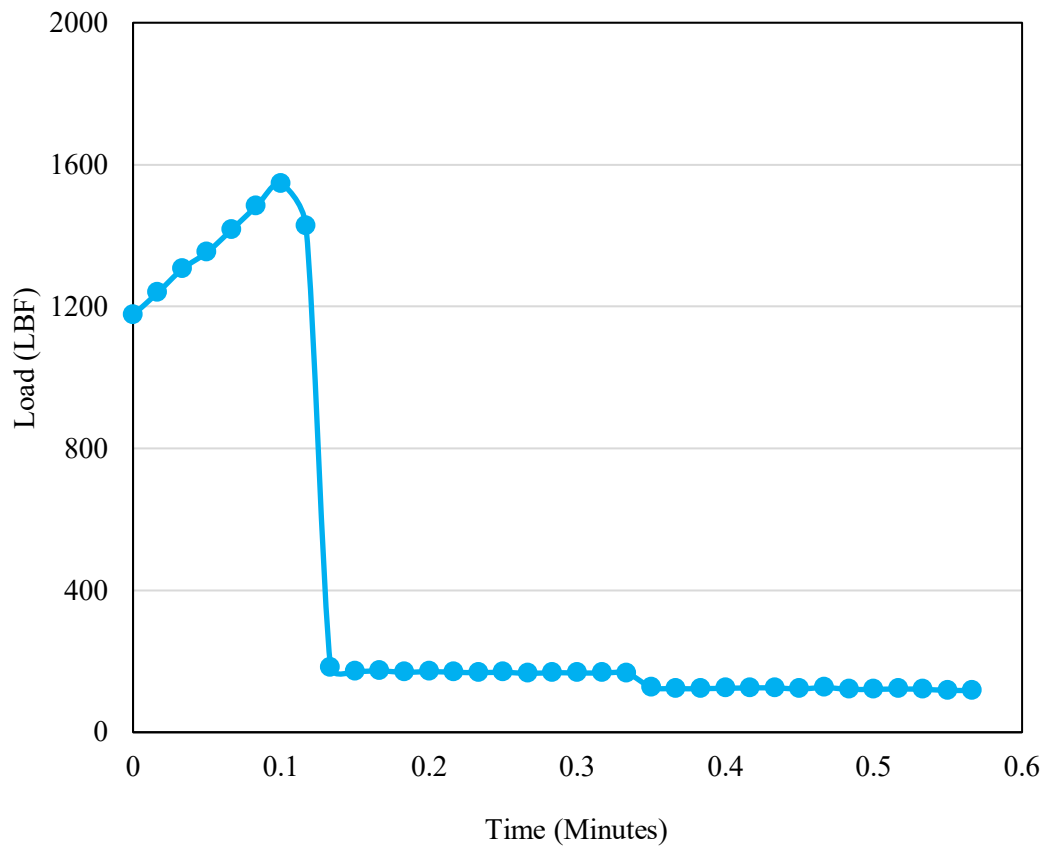


Figure 18. Load rate of the bonding measurement

Table 3. Bonding strength

Load (lbf)	Diameter (in)	Length (in)	Cross Sectional Area (in <sup>2</sup> )
1587	1.97	4	3.06



## 5. CONCLUSIONS

By studying the Novolac epoxy resin sealant, several conclusions were obtained. These findings are based on the results and analysis of the rheological measurements, curing results, blocking performance results, bonding strength, and compressive strength results. The main conclusions are summarized below:

- The behavior of Novolac epoxy resin is Newtonian with no or very low yield stress, which indicates that the material can be injected like water.
- The temperature plays a pivotal role when using thermosetting material, as increasing the temperature decreases the curing time. Obtaining a good estimation of the temperature where the material will be used is essential for better placement.
- The sealant has the ability to stop the leakage of water at cement fractures and channels to a differential pressure of up to 1800 psi.
- The compressive strength is 8931.3 psi after 24 hours of curing.
- The bonding strength is 1587 lbf after 24 hours of curing.

## ACKNOWLEDGEMENT

The author wishes to thank King Abdulaziz University (KAU) for granting him his scholarship.

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## SECTION

### 3. RESEARCH CONTRIBUTIONS AND BENEFITS

The main findings obtained from this study are as follows:

- Particles gel showed lower injectivity through small fractures and channels when compared to the solids-free Novolac epoxy resin sealant.
- The failure pressure of the particles gel is very low while the epoxy resin sealant resists differential pressure higher than 2000 psi without failing.
- The epoxy resin sealant develops good bonding with the cement in the fractures and the channels but the particles gel does not.
- The epoxy resin sealant was able to stop oil and water leakages while the particles gel was not able to mitigate oil and water leakage.

#### **4. RECOMMENDATION FOR FUTURE WORK**

The following are recommendations for future work to help address some issues related to the sealant materials:

- A new additives for the sealant material of must be developed and tested to increase the bonding strength with steel.
- Build a real wellbore experimental model that mimic downhole pressures and temperatures to get a better understanding of epoxy resin. The results of this model should be up scaled using finite element simulator.
- To develop a sealant material for high-temperature zones to avoid premature curing in the equipment.

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